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SUMMARY OF PROCEEDINGS

OF THE

FIFTIETH ANNUAL MEETING

ATLANTIC CITY, N. J., JUNE 16–20, 1947

The following Summary records the action taken by the Society at the Annual Meeting on the Recommendations Affecting Standards appearing in the annual reports of the standing committees. In the individual committee reports attention is called through an editorial footnote to any modifications of the recommendations or where the recommendations of the committee were not approved. In all other instances it is to be understood that the recommendations were approved as submitted by the committee. The new and revised standards and tentatives are published in the 1947 Supplement to Book of A.S.T.M. Standards, Parts I-A and I-B, II, and III-A and III-B. Except as noted, the reports and papers appear in this volume.

This summary of the Fiftieth Annual Meeting of the American Society for Testing Materials, held at Chalfonte-Haddon Hall, Atlantic City, N. J., June 16–20, 1947, is a record of the transactions of the meeting, including the actions taken on the various recommendations submitted by the standing committees. In all, nineteen technical sessions and a special business session were held.

The registered attendance of the meeting is as follows: Members present or represented, 1071; committee members, 469; guests, 246; total, 1786; ladies, 320.

The Proceedings are set forth session by session. There were 57 reports and 72 formal papers presented. The record with respect to each has been briefed, the recommendations in the reports having been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentatives and proposed revisions of existing standards, and as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentatives as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported as "adopted as standard" or "adopted as standard, revisions in" it is understood that this indicates approval of the Annual Meeting for reference to letter ballot of the Society. The various recommendations so recorded are included in the Society letter ballot.[1] The actions designated as "accepted as tentative" or "accepted as tentative, revisions in," are self-evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative.

While all of the items on the program are recorded under the particular session in which they were presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all the reports together with the page reference where the action thereon is recorded:

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[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, was canvassed on October 15, 1947, and all actions were approved.

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FIRST SESSION—OPENING SESSION; SYMPOSIUM ON LOAD TESTS OF BEARING CAPACITY OF SOILS

Opening Session

MONDAY, JUNE 16, 8 P.M.

SESSION CHAIRMAN: PRESIDENT ARTHUR W. CARPENTER

In opening the meeting President Arthur W. Carpenter called attention to the fact that this was the Fiftieth Annual Meeting of the Society. It was quite fitting that this was being recognized by having a very strong technical program which is open for participation on the part of all. He also called attention to the very many accomplishments of the Society as set forth in the report of the Board of Directors, particularly to the very successful District Meetings that had been held and to the activities of the technical committees which he considered the backbone of the Society.

Minutes of Forty-ninth Annual Meeting

The minutes of theForty-ninth Annual Meeting were distributed during the summer as a Summary of Proceedings. This summary is published in the 1946 *Proceedings*.[2] On motion, the minutes were approved.

Report of Board of Directors

In presenting the Board of Directors report, the Executive Secretary, C. L. Warwick, called attention first of all to the reports of the Administrative Committees that were appended to the report:

- Administrative Committee on Standards
- Administrative Committee on Research
- Administrative Committee on Papers and Publications
- Administrative Committee on Ultimate Consumer Goods
- Administrative Committee on Simulated Service Testing
- Administrative Committee on District Activities

These summarized the activities in a number of important fields. In addition, however, the Executive Secretary called attention to a reorganization that is being effected with respect to the review of methods of test through Committee E-1, the scope of activities of the Advisory Committee on Corrosion, and a contemplated change in the scope of activities of the Joint Committee on Effect of Temperature on the Properties of Metals so as to provide for the undertaking of standardization work in addition to its activities of a research nature.

The Board's report reviewed a number of important aspects of the Society's work including membership; meetings, both national and local; headquarters; publications; relations with other organizations such as the Federal Specifications Board; but more particularly the matter of finances. The auditors' report appended to the report of the Board includes a statement of the financial position of the Society for the last five years. This showed that the ratio of surplus to annual operating expenses at the current level is the lowest that it had been for some years—too low for sound operation. The report discusses in some detail the current finances of the Society as to its operation under the 1947 budget and considerations that enter into the 1948 and 1949 operations, leading to a recommendation for an increase in dues. In order to provide more adequately for discussion of such an important matter, it was proposed to make this a separate item of business in a special business session held for the purpose on Thursday morning.

There was one minor amendment of the By-laws however that was presented for action at this session, namely, an amendment of Article III, Section 2, changing the time for sending out and canvassing the returns of suggestions for the nominating committee so as to bring these dates in accord with the meeting dates established for the new Board of Directors. The revised Article III, Section 2, is to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

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[2] Summary of Proceedings of the Forty-ninth Annual Meeting, *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1 (1946).

> Not later than *July 1* [September 1] of each year the Executive Secretary shall notify every member of the Society that he has the right to recommend two members for appointment on the nominating committee. The procedure governing the transmission of such recommendations shall be designed to ensure secrecy as to individual authorship. A committee of tellers appointed by the President shall canvass the recommendations which have been received by the Executive Secretary prior to *August 15* [October 1], and shall transmit the returns to the Executive Secretary in time for presentation to the Board of Directors at its first regular meeting thereafter. . .

On motion, duly seconded, it was

VOTED that the proposed amendment be referred to letter ballot of the Society.[2a]

On motion, duly seconded, the report of the Board of Directors was accepted with the understanding that the proposed amendment of the By-laws with respect to the increase in dues would be presented at the business session on Thursday morning.

The Chair was then turned over to Mr. W. S. Housel who presided during the Symposium on Load Tests of Bearing Capacity of Soils as recorded below.

[For a record of the discussion and actions taken at the special business session of the Society held on Thursday morning with respect to the proposed amendment of Article VIII, Section 1 of the By-laws concerning dues, see pages 14 to 17.]

Symposium on Load Tests of Bearing Capacity of Soils[3]

MONDAY, JUNE 16, 8:30 P.M.

SYMPOSIUM SESSION CHAIRMAN: W. S. HOUSEL

The Symposium on Load Tests of Bearing Capacity of Soils consisted of the following six papers:

Field Loading Tests for the Evaluation of the Wheel Load Capacities of Airport Pavements—L. A. Palmer, presented from manuscript by the author.

Methods of Testing Soils for Runways and Foundations—Elwyn E. Seelye, W. D. Bailey, and S. D. Teetor, presented from manuscript by Mr. Bailey.

The Use of Load Tests in the Design of Flexible Pavements—W. H. Campen and J. R. Smith, presented from manuscript by Mr. Housel.

Field Bearing Tests Applied to Pavement Design—Robert R. Philippe, presented from manuscript by Mr. Frank M. Mellinger.

A Cyclic Load-Test Procedure—Jean E. Hittle and W. H. Goetz, presented from manuscript by Mr. Goetz.

A Canadian Investigation of Load Testing Applied to Pavement Design—Norman W. McLeod, presented from manuscript by the author.

[2a] The proposed amendment of Article III, Section 2 of the By-Laws was adopted by letter ballot which was canvassed on October 15, 1947, resulting in 890 affirmative and 4 negative votes.

[3] Issued as separate publication only.

SECOND SESSION—SYMPOSIUM ON MEASUREMENT OF ENTRAINED AIR IN CONCRETE

TUESDAY, JUNE 17, 9:30 A.M.

SESSION CHAIRMAN: A. T. GOLDBECK

Procedures for Determining the Air Content of Freshly-Mixed Concrete by the Rolling and Pressure Methods—Carl A. Menzel, presented from manuscript by the author.

Indiana Method for Measuring Entrained Air in Fresh Concrete—P. D. Miesenhelder, presented from manuscript by Mr. Joseph E. Gray.

The Measurement of Air Entrained in Concrete—John H. Swanberg and T. W. Thomas, presented from manuscript by Mr. Swanberg.

Comparison of Three Methods of Measuring Air Entrainment in Concrete—C. B. Schweizer, David Pirtz and Alexander Klein, presented from manuscript by Mr. R. E. Davis.

Measurement of Air Contents of Concrete by the Pressure Methods—H. W. Russell, presented by Mr. E. E. Bauer.

Analysis of Methods of Measuring Entrained Air in Concrete—W. A. Cordon and H. W. Brewer, presented from manuscript by Mr. W. H. Price.

The Pycnometer Method for Determining Entrained Air in Concrete—J. C. Pearson, presented by the author.

The Ohio Method of Determining the Amount of Air Entrained in Portland Cement Concrete—J. F. Barbee, presented from manuscript by the author.

THIRD SESSION—TECHNICAL PAPERS ON SOILS

TUESDAY, JUNE 17, 2 P.M.

SESSION CHAIRMAN: M. D. CATTON

The Use of the Maximum Principal Stress Ratio as the Failure Criterion in Evaluating Triaxial Shear Tests on Earth Materials—W. G. Holtz, presented from manuscript by the author.

Uplift Pressure on Bridge Foundations in Clay Revealed by Shear Tests—G. O. Kerkhoff and W. S. Housel, presented from manuscript by Mr. Kerkhoff.

Tests for Thermal Diffusivity of Granular Materials—William L. Shannon and Winthrop A. Wells, presented from manuscript by Mr. Shannon.

FOURTH SESSION—SYMPOSIUM ON INSULATING OIL[3a]

TUESDAY, JUNE 17, 2 P.M.

SESSION CO-CHAIRMEN: C. A. JOHNSON; L. B. SCHOFIELD

Advantages of an Inhibited Transformer Oil—T. E. Reamer and R. G. Larsen, presented by Mr. Larsen.

Oxidation Inhibitors in Electrical Insulating Oils—Leo J. Berberich, presented by the author.

Power Factor of Electrical Insulating Oils—Significance of and Methods of Testing Stability, J. C. Balsbaugh, presented by the author.

Serviceability Tests on Transformer Oil from the Viewpoint of the Maintenance Engineer—F. J. Pohnan, presented by the author.

FIFTH SESSION—TECHNICAL PAPERS ON CEMENT, LIME, AND CONCRETE

TUESDAY, JUNE 17, 8 P.M.

SESSION CHAIRMAN: FRANK H. JACKSON

Surface Areas of High Calcium Quicklimes and Hydrates—Howard R. Staley and Sidney H. Greenfeld, presented by Mr. W. C. Voss.

The Effect of Blends of Natural and Portland Cement on Properties of Concrete—A. G. Timms, W. E. Grieb, and George Werner, presented from manuscript by Mr. Timms.

Methods for the Determination of Soft Pieces in Aggregate—D. O. Woolf, presented from manuscript by the author.

The Chemical Reactions of Aggregates in Concrete—W. C. Hanna, presented from manuscript by the author.

The Effect of Sampling Errors on Unit Weight and Air Determinations in Concrete—J. C. Pearson and S. B. Helms, presented by Mr. Pearson.

[3a] Published in the ASTM BULLETIN, No. 149, December, 1947.

SIXTH SESSION—ELECTRODEPOSITED COATINGS AND METAL POLISHES

WEDNESDAY, JUNE 18, 9:30 A.M.

SESSION CHAIRMAN: SAM TOUR

Some of the Effects of Cadmium, Zinc, and Tin Plating on Springs—John R. Gustafson, presented from manuscript by the author.

Measurement of Metal Polish Performance[4]—Frank E. Clarke and Robert C. Adams, presented from manuscript by Mr. Clarke.

Committee B-8 on Electrodeposited Metallic Coatings:

Report presented by C. H. Sample, chairman.

Discussion on Exposure Test Panels:

Rating Exposure Test Panels of Decorative Electrodeposited Cathodic Coatings—W. A. Wesley, presented by the author.

Inspection of Exposure Test Panels with Nondecorative Electrodeposited Cathodic Coatings—H. A. Pray, presented by the author.

Inspection of Exposure Test Panels with Nondecorative Electrodeposited Anodic Coatings—Gustaf Soderberg, presented by the author.

SEVENTH SESSION—CEMENT, CONCRETE, BUILDING MATERIALS, ROAD AND PAVING MATERIALS

WEDNESDAY, JUNE 18, 1:30 P.M.

SESSION CHAIRMAN: J. L. MINER

Committee C-1 on Cement:

Report presented by F. H. Jackson, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Air-Entraining Portland Cement (C 175 - 46a T), with an additional revision[5] in Table II changing the limits on air content from 16 ± 4 per cent to 18 ± 3 per cent for Types IA and IIA cement.

Spec. for Portland Blast-Furnace Slag Cement (C 205 - 46 T).

Test for Air Content of Portland Cement Mortar (C 185 - 46 T), with the following additional revisions:[5]

Title.—Change to read: "Tentative Method of Test for Air Content of Air-Entraining Portland Cement Mortar."

Section 1.—Change to read: "This method of test is intended for determination of air content of air-entraining portland cement mortar under the conditions hereinafter specified."

Section 2(b).—Delete and make necessary redesignations of subsequent subsections.

Section 2(c).—Change to read: "A steel straight edge not less than $\frac{1}{16}$ in. nor more than $\frac{1}{8}$ in. in thickness."

Section 2(d).—Change to read: "A spatula with a metal blade 6 in. long and approximately $\frac{1}{2}$ in. wide with straight edges and a wooden handle."

Section 2(f).—Change to read: "A mixing bowl of 5 to 7 qt. capacity, made of stainless steel conforming in shape and dimensions to Fig. 1."

Section 2(h).—Change first sentence to read: "Glass graduates of 250-ml. capacities, with graduations in 2 ml., for measuring the mixing water, made to deliver the indicated volume at 20 C. (68 F.)."

Section 2(i).—Add the following as a second sentence: "The inner surface of the trough shall have an unplated, smooth polished finish." Change present second sentence to read: "The apparatus shall be constructed of materials and to the dimensions shown in Fig. 2." Delete from last sentence: "and free from set cement, dirt or grit." Add the following sentence at end of paragraph: "The base of the apparatus shall be fastened rigidly to a metal block weighing 10 ± 0.5 lb. during the flow determination." Add the following note at end of paragraph: "Note.—In the event that the balance point or weight of the trough is not within the prescribed tolerances, any weight added to or removed from the trough assembly shall be added to or removed from the stiffener web or the heel of the trough in such a manner that the change in weight shall be symmetrical about the long axis of the trough."

Section 5(a).—Change: "4-ml." to "3-ml."

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[4] Published in ASTM BULLETIN, No. 147, August, 1947.

[5] This recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee C-1 which ballot has been favorable.

Section 5 (b).—Revise the first sentence by deleting the words "continuous stirring," and add the following as a new second sentence: "No stirring action shall be employed except occasional sweeps of the gloved hand to remove mortar from sides of the bowl and bring it into a pile for the squeezing and kneading operation."

Section 5(c).—Delete from ninth sentence (line 9, column 1, 1946 Book of A.S.T.M. Standards, Part II, p. 1280) the words: "of the spatula."

Section 5(d).—Delete the third, fourth and fifth sentences. Replace by the following: "The mortar shall be placed in the 500-ml. measure in three equal layers, spading each layer thoroughly with the spatula around the inner surface of the measure. After the measure has been filled and spaded in the above prescribed manner, the sides of the measure shall be tapped lightly five times with the wooden handle of the spatula in order to preclude entrapment of extraneous air. Care shall be taken that no space is left between the mortar and the inner surface of the measure as a result of the spading operation."

Section 6.—Change to read: "The air content of the mortar shall be calculated from the following formula, which is based on use of the measure specified in Section 2(*a*) and on the batch proportions given in Section 5(*a*)."

Figure 1.—Replace by new Fig. 1 and add note as follows: "Bowls of this type should be available at restaurant supply houses."

Figure 2.—Present Fig. 1 revised. The revisions proposed in Fig. 2 are intended to secure appropriate tolerances in dimensions, clarify details that have been the subjects of recent questions, and effect certain desirable improvements.

Adopted as Standard, Revisions in:

Spec. for Portland Cement (C 150 – 46)

Methods of Chemical Analysis of Portland Cement (C 114 – 46)

Method of Test for Compressive Strength of Hydraulic-Cement Mortars (C 109 – 44), comprising the inclusion of the tentative revision issued in September, 1946.[5, 6]

Adopted as Standard:

Method of Test for Heat of Hydration of Portland Cement (C 186 – 44 T)

Committee C-7 on Lime:

Report presented by W. C. Voss, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Methods of Chemical Analysis of Limestone, Quicklime and Hydrated Lime (C 25 – 44)

Definitions of Terms Relating to Lime (C 51 – 44)

Committee C-8 on Refractories:

Report presented by J. D. Sullivan, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Castable Refractories for Boiler Furnaces and Incinerators

Accepted as Tentative, Revisions in:

Method of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes (C 133 – 39)

Definitions of Terms Relating to Refractories (C 71 – 46)

Spec. for Refractories for Heavy Duty Stationary Boiler Service (C 64 – 41)

Spec. for Refractories for Incinerators (C 106 – 41)

Spec. for Refractories for Moderate Duty Stationary Boiler Service (C 153 – 41)

Method of Test for Fireclay Refractories Under Load at High Temperatures (C 16 – 41)

Adopted as Standard:

Method of Test for Bonding Strength of Air-Setting Refractory Mortar (Wet Type) (C 198 – 45 T)

Method of Test for Refractoriness of Air-Setting Refractory Mortar (Wet Type) (C 199 – 45 T)

Method of Test for Thermal Conductivity of Insulating Fire Brick (C 182 – 45 T)

Method of Test for Thermal Conductivity of Refractories (C 201 – 45 T)

Method of Test for Thermal Conductivity of Fireclay Refractories (C 202 – 45 T)

Spec. for Air-Setting Refractory Mortar (Wet Type) for Boiler and Incinerator Service (C 178 – 45 T)

Spec. for Classification of Insulating Fire Brick (C 155 – 45 T)

Adopted as Standard, Revisions in:

Definitions of Terms Relating to Refractories (C 71 – 46)

Methods of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (C 38 – 45)

Methods of Panel Test for Resistance to Thermal and Structural Spalling (C 107 – 45)

Methods of Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 – 45)

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[6] 1946 Book of A.S.T.M. Standards, Part II, p. 1701.

Method of Panel Test for Resistance to Thermal and Structural Spalling of Fire Clay Plastic Refractories (C 180 - 45)
Spec. for Refractories for Malleable Iron Furnaces with Removable Bungs and for Annealing Ovens (C 63 - 41)
Spec. for Refractories for Heavy Duty Stationary Boiler Service (C 64 - 41)
Spec. for Refractories for Incinerators (C 106 - 41)
Spec. for Refractories for Moderate Duty Stationary Boiler Service (C 153 - 41)
Method of Test for True Specific Gravity of Burned Refractory Materials (C 135 - 40)
Spec. for Fireclay Plastic Refractories for Boiler and Incinerator Services (C 176 - 44)
Method of Test for Workability Index of Fireclay Plastic Refractories (C 181 - 45)
Spec. for Ground Fire Clay as a Mortar for Laying-Up Fireclay Brick (C 105 - 41)

Reaffirmation of Standard:

Classification of Fireclay Refractories (C 27 - 41)

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by Fred Hubbard, Vice-Chairman, and the following actions taken:

Accepted as Tentative:

Method of Test for Fundamental Transverse Frequency of Concrete Specimens for Computing Modulus of Elasticity (Sonic Method)[7]

Accepted as Tentative, Revisions in:

Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 - 44 T)
Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 - 46), jointly with Committee D-4 on Road and Paving Materials.
Spec. for Ready-Mixed Concrete (C 94 - 44)

Adopted as Standard:

Test for Surface Moisture in Fine Aggregate (C 70 - 44 T)

Adopted as Standard, Revisions in:

Test for Measuring Mortar-Making Properties of Fine Aggregate (C 87 - 46)
Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 - 46), jointly with Committee D-4.

Sanford E. Thompson Award:

Mr. Hubbard then introduced Mr. Sanford E. Thompson who made the sixth Sanford E. Thompson Award to William Lerch, Research Chemist, Research Laboratory of the Portland Cement Association, for his paper on "The Influence of Gypsum on the Hydration and Properties of Portland Cement Pastes" published in the 1946 *Proceedings*. This award was established in 1938 by Committee C-9 on Concrete and Concrete Aggregates as an annual token of recognition to the author or authors of a paper of outstanding merit on concrete and concrete aggregates presented at an annual meeting of the Society. In making the award, Mr. Thompson stated that Mr. Lerch's paper was selected because of its breadth of scope in research on the effect of gypsum in the different chemical compositions on cement and its value in practical concrete construction.

In accepting the certificate of the Sanford E. Thompson Award, Mr. Lerch expressed sincere appreciation of the recognition and honor conferred upon him and said that he was especially gratified to receive this certificate personally from Mr. Thompson.

Committee C-12 on Mortars for Unit Masonry:

Progress report presented, in the absence of the chairman, by F. O. Anderegg.

Committee C-14 on Glass and Glass Products:

Report presented, in the absence of the chairman, by L. C. Gilbert, and the following action taken:

Adopted as Standard:

Definition of the Term Glass (C 162 - 45 T)

Committee C-16 on Thermal Insulating Materials:

Report presented by Ray Thomas, chairman and the following actions taken:

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[7] This recommendation was accepted by unanimous consent, subject to favorable letter ballot of Committee C-9, which ballot has been favorable.

Accepted as Tentative:

Method of Test for Water Vapor Permeability of Sheet Materials Used in Connection with Thermal Insulation

Accepted as Tentative, Revisions in:

Definitions of Terms Relating to Thermal Insulating Materials (C 168 - 44), comprising definition of "molded-type pipe insulation"
Spec. for Mineral Wool Thermal Insulating Cement (C 195 - 45 T)
Spec. for Expanded or Exfoliated Vermiculite Thermal Insulating Cement (C 196 - 44 T)
Spec. for Diatomaceous Silica Thermal Insulating Cement (C 197 - 44 T)

Adopted as Standard, Revisions in:

Definitions of Terms Relating to Thermal Insulating Materials (C 168 - 44)

Committee C-18 on Natural Building Stones:

Report presented, in the absence of the chairman, by L. C. Gilbert, and the following actions taken:

Adopted as Standard, Revisions in:

Test for Absorption and Apparent Specific Gravity of Natural Building Stone (C 97 - 36)
Method of Flexure Testing of Natural Building Stone (Determination of Modulus of Rupture) (C 99 - 36), in the form of Test for Modulus of Rupture of Natural Building Stone.

Committee D-4 on Road and Paving Materials:

Report presented by F. H. Baumann, first vice-chairman, and the following actions taken:

Accepted as Tentative:

Test for Shear Strength of Flexible Road Surfaces, Subgrades and Fills by the Burggraf Shear Apparatus, jointly with Committee D-18 on Soils for Engineering Purposes.
Method of Testing Soil-Bituminous Mixtures, jointly with Committee D-18

Accepted as Tentative, Revisions in:

Test for Penetration of Bituminous Materials (D 5 - 25), in the form of a separate tentative method.
Methods of Testing Emulsified Asphalts (D 244 - 42), jointly with Committee D-8 on Bituminous Waterproofing and Roofing Materials, with an editorial change consisting of the addition of a section entitled "Outline of Methods" which contains a brief index of the tests included and pertinent conditions of each.
Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 - 46), jointly with Committee C-9 on Concrete and Concrete Aggregates.
Test for Hot Extraction of Asphaltic Materials and Recovery of Bitumen by the Modified Abson Procedure (D 762 - 44 T), comprising further refinement to the apparatus and reagents and clarifying the wording in the procedure.[8]

Adopted as Standard:

Spec. for Standard Sizes of Coarse Aggregate for Highway Construction (D 448 - 42 T)
Spec. for Tar (D 490 - 43 T)
Test for Cement Content of Soil-Cement Mixtures (D 806 - 44 T), jointly with Committee D-18.

Adopted as Standard, Revisions in:

Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 - 46), jointly with Committee C-9.
Spec. for Crushed Stone, Crushed Slag, and Gravel for Bituminous Concrete Base and Surface Courses of Pavements (D 692 - 42 T)

Committee D-8 on Bituminous Waterproofing and Roofing Materials:

Report presented by E. H. Berger, vice-chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Methods of Sampling Bituminous Materials (D 140 - 46 T), jointly with Committee D-4 on Road and Paving Materials.
Methods of Testing Asphalt Roll Roofing, Cap Sheets and Shingles (D 228 - 44 T)
Methods of Testing Emulsified Asphalts (D 244 - 42), jointly with Committee D-4.
Spec. for Asphalt for Dampproofing and Waterproofing (D 449 - 42 T)

Adopted as Standard, Revisions in:

Spec. for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 226 - 44)
Spec. for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 227 - 44)

Adopted as Standard:

Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (D 146 - 44 T), with editorial changes.

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[8] This recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee D-4, which ballot has been favorable.

Spec. for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-Up Roofs (D 250 - 44 T)

Spec. for Asphalt-Saturated and Coated Asbestos Felts for Use in Constructing Built-Up Roofs (D 655 - 44 T)

Committee D-18 on Soils for Engineering Purposes:

Report presented by E. J. Kilcawley, chairman, and the following actions taken:

Accepted as Tentative:

Test for Shear Strength of Flexible Road Surfaces, Subgrades, and Fills by the Burggraf Shear Apparatus,[9] jointly with Committee D-4 on Road and Paving Materials.

Method of Testing Soil-Bituminous Mixtures,[9] jointly with Committee D-4.

Adopted as Standard:

Test for Cement Content of Soil-Cement Mixtures (D 806 - 44 T), jointly with Committee D-4.

It was announced that Committee D-18 planned to recommend to the Standards Committee for publication as tentative a new Method of Test for Field Determination of Density of Soil in Place, and a revision of the Tentative Method of Test for Moisture-Density Relations of Soils (D 698 - 42 T), the latter subject to concurrence of Committee D-4. These items had been referred to in the report as preprinted.

Committee E-5 on Fire Tests of Materials and Construction:

Report presented by S. H. Ingberg, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Methods of Fire Tests of Building Construction and Materials (E 119 - 41)

Method of Test for Combustible Properties of Wood by the Fire-Tube Test Method (E 69 - 46 T)

Committee E-6 on Methods of Testing Building Constructions:

Report presented by L. J. Markwardt, chairman, which included announcement of the acceptance by the Administrative Committee on Standards of the new Tentative Methods of Conducting Strength Tests of Panels for Building Constructions (E 72 - 47 T).

EIGHTH SESSION—SYMPOSIUM ON SYNTHETIC LUBRICANTS, REPORTS AND TECHNICAL PAPERS

WEDNESDAY, JUNE 18, 1:30 P.M.

SESSION CHAIRMAN: E. H. ERCK

SESSION SECRETARY: J. C. GENIESSE

Committee D-16 on Industrial Aromatic Hydrocarbons:

Report presented by D. F. Gould, chairman, and the following actions taken:

Accepted as Tentative:

Test for Thiophene in Benzene

Adopted as Standard:[10]

Spec. for Nitration Grade Benzene (D 835 - 45 T)

Spec. for Industrial Grade Benzene (D 836 - 46 T)

Spec. for Industrial 90 Benzene (D 837 - 45 T)

Spec. for Refined Solvent Naphtha (D 838 - 45 T)

Spec. for Crude Light Solvent Naphtha (D 839 - 45 T)

Spec. for Crude Heavy Solvent Naphtha (D 840 - 45 T)

Spec. for Nitration Grade Toluene (D 841 - 45 T)

Spec. for Industrial Grade Toluene (D 842 - 45 T)

Spec. for Nitration Grade Xylene (D 843 - 45 T)

Spec. for Industrial Grade Xylene (D 844 - 45 T)

Spec. for Five-Degree Xylene (D 845 - 45 T)

Spec. for Ten-Degree Xylene (D 846 - 45 T)

Test for Acidity of Benzene, Toluene, Xylenes, Solvent Naphthas, and Similar Industrial Aromatic Hydrocarbons (D 847 - 45 T)

Test for Acid Wash Color of Benzene, Toluene, Xylenes, Refined Solvent Naphthas, and Similar Industrial Aromatic Hydrocarbons (D 848 - 45 T)

Test for Copper Corrosion of Industrial Aromatic Hydrocarbons (D 849 - 45 T)

Test for Distillation of Industrial Aromatic Hydrocarbons (D 850 - 45 T)

Test for Paraffins in Industrial Aromatic Hydrocarbons (D 851 - 45 T)

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[9] These recommendations were accepted, subject to favorable letter ballot of Committee D-18, which ballot has been favorable.

[10] These recommendations, not preprinted, were accepted by unanimous consent, subject to favorable letter ballot of Committee D-16, which ballot has been favorable.

Test for Solidifying Point of Benzene (D 852 - 45 T)
Test for Color, and Hydrogen Sulfide and Sulfur Dioxide Content (Qualitative) of Industrial Aromatic Hydrocarbons (D 853 - 45 T)

Committee D-3 on Gaseous Fuels:

Report presented, in the absence of the chairman, by P. J. Smith.

Committee D-2 on Petroleum Products and Lubricants:

Report presented by C. Dantsizen, chairman and the following actions taken:

Adopted as Standard, Revisions in:

Test for Knock Characteristics of Motor Fuels (D 357 - 46)
Methods of Analysis of Grease (D 128 - 40)
Test for Oil Content of Paraffin Wax (D 721 - 44)
Test for Cloud and Pour Points (D 97 - 39)
Definitions of Terms Relating to Petroleum (D 288 - 39)

It was announced that the large number of recommendations referred to in the report as preprinted covering various new tentatives and revisions in existing tentatives would be presented to the Society through the Standards Committee subsequent to the Annual Meeting. In addition to the recommendations mentioned in the preprinted report, the committee also plans to recommend the reversion to tentative and revision of the Standard Method of Test for Water and Sediment in Petroleum Products by Means of Centrifuge (D 96 - 46), and an editorial revision of Standard D 341 covering a new Viscosity Temperature Chart E covering the temperature range from −100 to +450 F. and a viscosity range of 2.0 to 20,000,000 centistokes.

Sectional Committee Z11 on Petroleum Products and Lubricants:

Report, not preprinted, presented from manuscript by P. J. Smith, secretary.

Paper:

The following paper was presented:
Polarographic Determination of Tetraethyl Lead in Gasoline—Richard Borup and Harry Levin, presented from manuscript by Mr. Levin.

Symposium on Synthetic Lubricants:[3]

The "Ucon" Synthetic Lubricants and Hydraulic Fluids—J. M. Russ, presented from manuscript by the author.
Synthetic Lubricants from Diesters—H. R. Stringer and F. J. Glavis, presented from manuscript by Mr. Glavis.
Synthetic Lubricants for Military Aircraft—C. C. Singleterry, presented from manuscript by the author.

NINTH SESSION—ROUND TABLE DISCUSSION ON SPEED OF TESTING

WEDNESDAY, JUNE 18, 1:30 P.M.
SESSION CHAIRMAN: P. G. MCVETTY

This Round Table Discussion on Speed of Testing was sponsored by the Section on Effect of Speed of Testing, of Committee E-1 on Methods of Testing, preliminary to a more formal Symposium at the 1948 Annual Meeting.

In opening the session Mr. McVetty reviewed briefly the reasons for arranging this program and explained that it was for the purpose of uncovering undesirable features in existing speed specifications and to determine the best procedure for correcting them.

The subject was then introduced by Prof. H. F. Moore who presented a prepared discussion entitled "Effect of Speed of Testing on Strength and Ductility of Metals."

There followed interesting oral discussion by individuals representing various A.S.T.M. technical committees presenting a review of this problem as regards the speed of testing materials under the jurisdiction of these committees, including Messrs. L. H. Fry, R. H. Heyer, G. R. Gohn, N. H. Murzda, F. M. Howell, L. J. Markwardt and A. G. H. Dietz.

The meeting was concluded by general discussion by those present.

TENTH SESSION—MARBURG LECTURE; RICHARD L. TEMPLIN AWARD

WEDNESDAY, JUNE 18, 4:30 P.M.

SESSION CHAIRMAN: PRESIDENT ARTHUR W. CARPENTER

In introducing the Twenty-first Marburg Lecturer, President Carpenter recalled that the purpose of the lecture was to have described at annual meetings of the Society, by leaders in their respective fields, outstanding developments in the promotion of knowledge of engineering materials. Established as a means of emphasizing the importance of one of the functions of the A.S.T.M. in the promotion of knowledge of materials, the lecture honors and perpetuates the memory of Edgar Marburg, first Secretary of the Society, who placed its work on a firm foundation and through his development of the technical programs brought wide recognition to the A.S.T.M. as a forum for the discussion of properties and tests of engineering materials.

President Carpenter then presented Mr. W. C. Voss, Head, Department of Building and Construction, Massachusetts Institute of Technology, who delivered the lecture on "Engineering Laminates."[11] The talk covered a discussion of engineering laminates which as conceived by most investigators should be broadly construed as including metals, wood, plastics, and fabrics, combined each to each and each to the other. He pointed out that the problems underlying the inhomogeneity of the materials used in the production of engineering laminates have their basis in the fundamental sciences and still pose a vexing problem to the profession.

President Carpenter, in expressing appreciation to Mr. Voss for his instructive and very interesting lecture on a subject of particular interest to the A.S.T.M., presented to him on behalf of the Society, the Edgar Marburg Lecture Certificate.

Richard L. Templin Award:

The chairman recognized W. A. Evans, chairman of the Committee on the Richard L. Templin Award. Mr. Evans presented Mr. F. B. Quinlan, Metallurgical Section, Works Laboratory, General Electric Co., to whom the second Richard L. Templin Award was made for his paper on "Pneumatic Fatigue Machines" published in the 1946 *Proceedings* and in the Symposium on Parts and Assemblies. Mr. Evans stated that the purpose of the award is to stimulate research in the development of testing methods and apparatus, to encourage the presentation to the Society of papers describing new and useful testing procedures and apparatus, and to recognize meritorious efforts of this character. The award was established in 1945 when the Executive Committee received a gift from Richard L. Templin to be used for a prize award for papers describing new testing methods and apparatus.

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[11] See p. 449; also available as a separate publication.

ANNUAL MEETING DINNER

WEDNESDAY, JUNE 18, 7 P.M.

TOASTMASTER: PAST-PRESIDENT W. H. FULWEILER

The Annual Meeting Dinner was attended by 293 members and guests.

At the conclusion of the dinner Mr. Fulweiler recalled that on this occasion the Society was celebrating its Fiftieth Annual Meeting, the first having been held in 1898 in Philadelphia under the auspices of the American Section of the International Association for Testing Materials.

Election of Officers:

The toastmaster then recognized A. O. Schaefer, chairman of the Committee of Tellers, who reported results of the letter ballot on election of officers. Of the 1585 ballots cast, the results were as follows:

For President, to serve for one year:
  T. A. Boyd, 1585 votes.
For Vice-President, to serve for two years:
  J. G. Morrow, 1585 votes.
For Directors, to serve for three years:
  T. S. Fuller, 1585 votes.
  Everett G. Ham, 1585 votes.
  J. J. Laudig, 1585 votes.
  H. L. Maxwell, 1585 votes.
  L. J. Trostel, 1585 votes.

The toastmaster then introduced the newly elected members of the Board of Directors, requesting that they rise as their names were called.

The toastmaster then introduced the Vice-President-Elect, J. G. Morrow, who expressed appreciation for the honor conferred on him, especially since he was the first member of the Society from Canada to be elected an officer of the Society. He expressed the hope that this would further serve to cement the cooperative relations between the two countries in the field of standardization.

The toastmaster then introduced the President-Elect, T. A. Boyd. Mr. Boyd, in expressing sincere appreciation for the honor conferred on him, said that he realized fully the many responsibilities entailed, particularly at this time in view of the many problems and new projects that will need to be considered as the result of developments during the war.

The toastmaster also introduced Mr. G. C. MacDonald who had been appointed a Director to fill the unexpired term of Mr. A. J. Chameroy.

Award of Honorary Memberships:

President Carpenter then made awards of honorary memberships to the following three members of the Society: F. Malcolm Farmer, Arno Carl Fieldner, and Hervey S. Vassar, after they were presented, respectively, by Past-Presidents J. R. Townsend, P. H. Bates, and Dean Harvey. Each past-president, in presenting the recipient, reviewed briefly his accomplishments and directed particular attention to the contributions or services that he had rendered to the Society. Each of the recipients of honorary membership in turn expressed appreciation of the honor conferred upon him.

Presidential Address:

The toastmaster then presented the President, Arthur W. Carpenter, who delivered the annual presidential address entitled "Our Stake in Materials Progress."[12]

Recognition of Forty-Year Members:

The toastmaster recognized the Executive Secretary, C. L. Warwick, who announced that in accordance with action taken by the Board of Directors, certificates were being awarded this year to eleven individuals and three companies who had attained 40 years of membership in the Society. Presentation of the certificates was then made by the Executive Secretary to the following:

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[12] See p. 32.

Herbert Abraham, President, The Ruberoid Co., New York, N. Y.
E. H. Beckstrand, Professor of Mechanical Engineering (Retired), University of Utah, Salt Lake City, Utah.
Henry C. Boynton, Consulting Metallurgist, John A. Roebling's Sons Co., Trenton, N. J.
Alexander J. Christie, Cynwyd, Pa.
Thomas R. Lawson, Technical Representative, The Barrett Division, Allied Chemical and Dye Corp., New York, N. Y.
C. P. Marsh, Formerly Assistant Engineer (Retired), New York Central Railroad Co., New York, N. Y.
Raymond G. Osborne, Testing Engineer, Raymond G. Osborne Testing Laboratories, Los Angeles, Calif.
Reading Company, Reading, Pa.
Rensselaer Polytechnic Institute, Troy, N. Y.
Harry A. Schwartz, Manager of Research, National Malleable and Steel Castings Co., Cleveland, Ohio.
John Fairfield Thompson, Executive Vice-President, The International Nickel Co., Inc., New York, N. Y.
Percy H. Walker, Associate Chemist (Retired), National Bureau of Standards, Washington, D. C.
Morton Owen Withey, Professor of Mechanics, University of Wisconsin, Madison, Wis.
The Youngstown Sheet and Tube Co., Youngstown, Ohio.

Address by Dr. Roy K. Marshall:

The toastmaster then presented Roy K. Marshall, Ph.D., of the Franklin Institute and The Philadelphia Evening Bulletin, as the guest speaker. Dr. Marshall then presented an address entitled "Materials Problems in the Atomic Age."

SPECIAL BUSINESS SESSION

THURSDAY, JUNE 19, 9 A.M.

SESSION CHAIRMAN: PRESIDENT A. W. CARPENTER

To supplement the discussion of finances set forth in the report of the Board of Directors in connection with the proposed amendment to the By-laws to increase the membership dues, a statement had been distributed to members attending the meeting, outlining the salient considerations in recommending an increase in dues. President Carpenter called on Executive Secretary Warwick, who, in presenting this statement placed before the meeting the proposed amendment of Article VIII, Section 1, to read as follows:

> The fiscal year shall commence on the first day of January. The annual dues, payable in advance, shall be as follows: For Individual Members, $18; for Company Members, $50; for Sustaining Members, $150; for Junior Members, $9; for Student Members, $2. Honorary Members shall not be subject to dues.

This would increase the dues of individual members from $15 to $18; company members from $30 to $50; sustaining members from $100 to $150; junior members from $7.50 to $9; and student members from $1.50 to $2.

Mr. Warwick, referring to the Board of Directors' report, called attention to the deficit of $28,000 that had been incurred in 1946, and the probable deficit in 1947—a year in which, because of the triennial nature of the budgeting operations necessitated by the triennial publication of the Book of Standards, there should be instead a substantial surplus for application to 1948 and 1949 operations. These deficits, of course, have come about through the greatly increased cost of operations, especially printing and staff personnel; in fact, practically all expenses had risen sharply during the past three years. The Board estimates that with income in sight at present dues, and maintaining current levels of activity, there would be operating deficits in each of the next two years of from $30,000 to $40,000. It was not felt that other sources of revenue could be increased materially beyond that already accomplished, and even so, it was not considered too sound to place the Society's revenue on the basis where receipts from dues played an ever smaller part.

After the amendment had been placed formally before the meeting, the subject was thrown open for discussion which was participated in by G. H. von Fuchs, R. C. Adams, H. A. Eysenbach, J. T. MacKenzie, R. D. Stiehler, Past-President J. R. Townsend, and Vice-President T. A. Boyd. President Carpenter, in participating in this discussion, called attention to the fact that this was the first increase in dues since 1928 at which time the dues for company members had been increased, individual dues having stood at the present rate since 1916.

Mr. von Fuchs, speaking as an individual member, wished to have consideration given to a plan whereby the dues for individuals would be lowered to say $10 rather than raised, a plan similar to that followed by the American Chemical Society, under which the member for his nominal dues would receive only a limited amount of publications, all other publications being on a selected basis for which the member would be charged. He felt this would bring about greatly increased membership and thus better dissemination of information concerning the Society and its publications. The member, he thought, did not need all of the publications which he now receives on his membership.

It was pointed out by President Carpenter and Vice-President Boyd that careful consideration had been given to such a proposal and it was deemed unwise to change the present basis of membership. Although some greater selectivity might need to be made in publications, the fact remains that the expected economies are not necessarily found in decreasing editions of the individual publications since a great amount of work needs to be accomplished in putting a book on press whether the edition be one thousand or five thousand.

Mr. Adams raised the question of the cost of an individual member to the Society. He made the point that so far as those individual members are concerned who are active in committee work and who attend meetings, the dues themselves are relatively minor expenses; that the time and effort and actual money contributed by working individual members far exceeds the amount of dues and that there are many individual members who do not make that contribution but receive the same benefits from the Society. He would wish to know the cost to the Society of the services that are automatically provided to these individuals.

Executive Secretary Warwick was called upon to present information in this connection. He stated that an analysis had been made of the mechanical costs (not including any overhead expense) of printing publications a member receives on his dues. These publications include the annual *Proceedings*, one part of the Book of Standards, an Index to Standards, the ASTM BULLETIN, the Year Book, if requested, and a set of preprints if requested. The cost of printing the Proceedings per member is $4; one part of the Book of Standards, $3.60; the Index to Standards, 30 cents; the Year Book, $1; and the cost of a set of preprints, $2.50. The Bulletin is not included since the mechanical costs are offset by advertising.

The disbursements and receipts for 1946 had been analyzed and placed in two categories, the one representing estimated costs, including overhead, on publications that are sold either to members outside their dues or to nonmembers; and the other, the costs of publications and services to members on their dues. In this latter category were included all the publications that are printed for distribution to the membership, the proportion of the salaries that goes into membership operations, general office expenses, work of the committees, meetings, headquarters occupancy, and miscellaneous items. This analysis, as set forth in the accompanying Table I, was thrown on the screen. In 1946 the Society spent $41.25 per member for services to members, and $14.45 per member for publications for sale, making a total of $55.70 per member. Dues received per member averaged $21.45. Adding in all charges, the members contributed through dues or fees, $25.80 per member. Other sources such as advertisement in Bulletin, income from investments, income from exhibits, etc., contributed $4.80 per member, making the total received per member $30.60, so that the deficit per member proved to be $10.65. On the other hand, publication sales brought in $20.55 per member, which showed a surplus for publication sales of $6.10 above expense, which was responsible for the net deficit being only $4.55 per member.

TABLE I.—1946 INCOME AND DISBURSEMENTS "PER MEMBER."

(BASED ON MEMBERSHIP OF 6100)

	"General" Operations Including Services to Members	For Publications for Sale	Total
Disbursements			
Publications	13.85	7.20	
Salaries	14.87	5.20	
General Office Expense	4.50	1.85	
Committees, Meetings	3.76	0.20	
Headquarters Occupancy Exp.	1.84	....	
Miscellaneous	2.43	....	
Total	41.25	14.45	55.70
Receipts			
Dues	21.45[a]		
Entrance Fees	0.95		
Meeting Registration Fees	0.62		
Charges for Parts of Book of Standards	2.78		
	25.80		
Other Sources: Advertising, Dividends, Exhibit, Misc.	4.80	20.55	
Total	30.60	20.55	51.15
Deficit in "General Operations"	10.65		
Surplus in Publication Sales		6.10	
Net Deficit in Operations			4.55

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[a] Made up approximately as follows: $15 from 4200 individual members; $30 from 1600 company members; $100 from 200 sustaining members. Industry as represented by company and sustaining members contributes about 52 per cent of the income from dues.

Mr. Eysenbach, speaking as an individual member, expressed the view that it was not a matter of value but competition for the dollar, and in accepting the proposed modification he felt that the members should give careful thought to extending the usefulness of the Society in the smaller organizations, thus bringing the work of the Society to the attention of all individuals who might benefit.

Past-President Townsend, in addition to calling attention to the fact that the proposed increase in dues in the case of individuals was really nominal (all such expenses including expenses incurred in attending meetings are deductible from income tax), expressed the view that the important thing was that an organization such as the American Society for Testing Materials should be supported as a place where an individual interested in materials might meet and exchange knowledge and to make technical contributions, and in association with his fellows carry forward the standardization work and development of methods of testing. He felt that even without publications the experience and stimulation secured was of sufficient importance.

Mr. Stiehler expressed the viewpoint of a government employee who frequently attends meetings at his own expense to whom the matter of dues is one of some importance.

After some further discussion with respect to the merits of alternate plans for establishing dues, also whether this was an appropriate time for making the change, a motion to refer the proposed amendment to letter ballot of the Society[13] was adopted with one dissenting vote, that recorded by Mr. von Fuchs.

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[13] The proposed amendment of Article VIII, Section 1, of the By-Laws was adopted by letter ballot which was canvassed on October 15, 1947, resulting in 849 affirmative and 56 negative votes.

ELEVENTH SESSION—TECHNICAL PAPERS ON PLASTIC DEFORMATION OF METALS

THURSDAY, JUNE 19, 10 A.M.

(Adjourned at 12:30 P.M. and reconvened at 2 P.M.)

SESSION CHAIRMAN: VICE-PRESIDENT R. L. TEMPLIN

An Experimental Study of the Propagation of Plastic Deformation Under Conditions of Longitudinal Impact—**P. E. Duwez and D. S. Clark**, presented by Mr. Clark.

The Velocity Aspect of Tension Impact Testing—William H. Hoppmann, presented by the author.

Plastic Flow of a Magnesium Alloy Under Biaxial Stresses—D. M. Cunningham, E. G. Thomsen, and J. E. Dorn, presented by Mr. Cunningham.

Influence of Plastics Extension and Compression on the Fracture Stress of Metals—D. J. McAdam, Jr., G. W. Geil, and W. H. Jenkins, presented by Mr. McAdam.

Intercrystalline Cohesion and the Stress-Rupture Test—H. H. Bleakney, presented by the author.

The Progress of Failure in Metals as Traced by Changes in Magnetic and Electrical Properties—P. E. Cavanagh, presented by the author.

A Study of the Transition from Shear to Cleavage Fracture in Mild Steel—H. E. Davis, E. R. Parker and Alexander Boodberg, presented from manuscript by Mr. Max Gensemer.

Physical Characteristics of Steel for Tubular Products—Arthur B. Wilder, presented from manuscript by the author.

A New Type of Magnetic Flaw Detector—Carlton H. Hastings, presented by title only.[14]

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[14] Paper presented in full at a meeting of Committee E-7 on Radiographic Testing held on Monday, June 16, at 8 P.M.

TWELFTH AND THIRTEENTH SESSIONS—SYMPOSIUM ON RUBBER TESTING[3]

Twelfth Session

THURSDAY, JUNE 19, 10 A.M.
SESSION CHAIRMAN: SIMON COLLIER

The Significance of Voluntary Standards and Their Status in the Rubber Industry—A. W. Carpenter, presented from manuscript by the author.

Functions of Rubber Reserve, Past, Present and Projected—W. R. Hucks, presented from manuscript by the author.

Development of Methods of Chemical Analysis of Synthetic Rubber—Willard P. Tyler and T. Higuchi, presented from manuscript by Mr. Tyler.

Development and Improvement in Methods of Stress-Strain Testing of Rubber—J. W. Schade and F. L. Roth, presented from manuscript by Mr. Roth.

Thirteenth Session

THURSDAY, JUNE 19, 2 P.M.
SESSION CHAIRMAN: H. E. OUTCAULT

Development and Standardization of Tests for Evaluating Processibility of Rubber—Rolla H. Taylor, J. H. Fielding, and M. Mooney, presented from manuscript by Mr. Taylor.

Standardization of Testing and Inspection in Government Synthetic Rubber Plants—Ludwig Meuser, Robert D. Stiehler, and R. W. Hackett, presented from manuscript by Mr. Meuser.

Testing and Grading of Wild and Plantation Rubbers—Norman Bekkedahl, presented from manuscript by the author.

The Use of Statistical Methods in Rubber Evaluation—Marian M. Sandomire, presented from manuscript by the author.

FOURTEENTH SESSION—NON-FERROUS METALS

THURSDAY, JUNE 19, 8:00 P.M.
SESSION CHAIRMAN: J. H. FOOTE

Committee B-1 on Wires for Electrical Conductors:

Report presented by E. H. Kendall, secretary, and the following actions taken:

Adopted as Standard, Revisions in:

Spec. for Hard-Drawn Copper Wire (B 1 – 40)
Spec. for Medium-Hard-Drawn Copper Wire (B 2 – 40)
Spec. for Hard-Drawn Copper Alloy Wires (B 105 – 39)

Accepted as Tentative, Revisions in:

Spec. for Rope-Lay-Stranded Copper Conductors Having Concentric-Stranded Members, for Electrical Conductors (B 173 – 45 T)

The committee also reported that its reorganization had been completed during the past year and seven subcommittees constituted of which four had just held meetings, and a program of work for the coming year had been outlined and assigned to task groups.

Committee B-2 on Non-Ferrous Metals and Alloys:

Report presented by E. E. Thum, chairman, and the following action taken:

Adopted as Standard:

Spec. for Oxygen-Free Electrolytic Copper Wire Bars, Billets, and Cakes (B 170 – 44 T)

In presenting the report, mention was made of the formation during the year, on the recommendation of the Non-Ferrous Coordinating Committee, of a Joint B-1, B-2, B-5 Committee on the Classification of Copper. This joint committee had held several meetings, and its work progressed to such a point that the classification, which has been a long standing project, should take final form and be available by the end of the year.

Paper:

The following paper was presented:

Hardness Conversion Chart for Nickel and High Nickel Alloys—F. P. Huston, presented from manuscript by the author.

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented by A. W. Tracy, secretary, and the following action taken:

Editorial Change Accepted in:

Method of Alternate Immersion Corrosion Testing of Non-Ferrous Metals (B 192 - 44 T)

Committee B-4 on Electrical Heating, Resistance, and Related Alloys:

Report presented by J. W. Harsch, chairman, and the following actions taken:

Accepted as Tentative:

Method of Measuring Residual Stress in Cylindrical Metal-to-Glass Seals
Method of Testing Fine Round and Flat Wire for Electronic Devices

Withdrawal of Tentatives:

Method of Testing Nickel and Nickel-Alloy Wire and Ribbon for Electronic Tube Filaments (B 118 - 42 T)
Method of Testing Lateral Wire for Grids of Electronic Devices (B 156 - 42 T)

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented by V. P. Weaver, secretary, and the following actions taken:

Accepted as Tentative:

Method of Tension Testing of Copper and Copper-Alloy Rods, Bars, and Shapes

Accepted as Tentative, Revisions in:

Spec. for Cartridge Brass Sheet, Strip, Plate, Bar, and Disks (B 19 - 46 T), with editorial changes
Spec. for Naval Brass Rods, Bars, and Shapes (B 21 - 46a T)
Spec. for Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 - 46 T)
Spec. for Brass Sheet and Strip (B 36 - 46 T), with editorial changes
Spec. for Seamless Copper Tubes (B 75 - 46a T)
Spec. for Leaded Brass Sheet and Strip (B 121 - 46 T)
Spec. for Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 - 46a T), with the correction that Table IV be revised to read the same as Table III of B 103.
Spec. for Cartridge Brass Cartridge Case Cups (B 129 - 46 T)
Spec. for Gilding Metal Strip (B 130 - 46 T)
Spec. for Gilding Metal Bullet Jacket Cups (B 131 - 46 T)
Spec. for Copper Rods, Bars, and Shapes (B 133 - 46a T)
Spec. for Miscellaneous Brass Tubes (B 135 - 46a T)
Spec. for Phosphor Bronze Rods, Bars, and Shapes (B 139 - 46a T)
Spec. for Aluminum Bronze Rods, Bars, and Shapes (B 150 - 46a T)
Spec. for Copper-Nickel-Zinc Alloy Rod, Bar, and Wire (B 151 - 46a T)
Spec. for Copper Sheet, Strip, and Plate (B 152 - 46 T)
Spec. for Phosphor Bronze Wire (B 159 - 46a T)
Spec. for Aluminum Bronze Sheet and Strip (B 169 - 46 T)
Spec. for Copper Bus Bars, Rods and Shapes (B 187 - 46 T)
Spec. for Copper Bus Pipes and Tubes (B 188 - 46 T)
Spec. for Beryllium-Copper Alloy Strip (B 194 - 46a T)
Spec. for Beryllium-Copper Alloy Strip, Special Grade (B 195 - 46 T)
Spec. for Beryllium-Copper Alloy Rod and Bar (B 196 - 46a T)
Spec. for Beryllium-Copper Alloy Wire (B 197 - 46a T), with the added change that the revision indicated for Section 7 include "and delete the present elongation values."
Spec. for Copper-Nickel-Zinc Alloy Wire (B 206 - 46a T), with editorial changes.

Adopted as Standard, Revisions in:

Spec. for Copper Plates for Locomotive Fireboxes (B 11 - 46)
Spec. for Copper Bars for Locomotive Staybolts (B 12 - 46)
Spec. for Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 - 46)
Spec. for Copper Pipe, Standard Sizes (B 42 - 46)
Spec. for Red Brass Pipe, Standard Sizes (B 43 - 46)
Spec. for Seamless Copper Tubing, Bright Annealed (B 68 - 46)

Spec. for Copper Water Tube (B 88 - 46)

Spec. for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 - 46) with the following corrected revisions for Sections 3, 5 (*a*) and 6:

Section 3.—Designate the present Section as Paragraph (*b*) and revise to read, "The plate and sheet shall be manufactured by hot rolling and annealing or by cold rolling and annealing." Add a new Paragraph (*a*) to read as recommended for Section 3 (*a*) in Specifications B 19.

Section 5 (*a*).—Revise to read "A sample shall be taken from each 5000 lb. or fraction thereof. The sample shall consist of equal quantities taken from three pieces. If the lot be of less than three pieces, a sample shall be taken from each piece."

Section 6.—Change the yield strength value from 29,000 to 18,000 psi.

Spec. for Copper-Silicon Alloy Sheet and Strip for General Purposes (B 97 - 46), with the following corrected revisions for Section 3:

Section 3.—Revise the title to read "Manufacture." Delete Paragraph (*b*), designating the present Paragraph (*a*) as (*b*) and revise to read, "The material shall be manufactured by hot rolling or cold rolling followed by such annealing as may be required. Add a new Paragraph (*a*) to read as recommended for Section 3 (*a*) in Specifications B 19.

Spec. for Copper-Silicon Alloy Rods, Bars and Shapes (B 98 - 46)

Spec. for Copper-Silicon Alloy Wire for General Purposes (B 99 - 46)

Spec. for Rolled Copper-Alloy Bearing and Expansion Plates for Bridge and Other Structural Uses (B 100 - 46), with the following corrected revisions for Section 5 (*a*):

Section 5 (*a*).—Change report text as preprinted to read the same as given above for Section 5(*a*) of Specifications B 96, except make the lot size 2000 lb. instead of 5000 lb.

Spec. for Phosphor Bronze Sheet and Strip (B 103 - 46)

Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 46)

Spec. for Copper-Base Alloy Forging Rods, Bars and Shapes (B 124 - 46)

Spec. for Brass Wire (B 134 - 46)

Spec. for Manganese Bronze Rods, Bars, and Shapes (B 138 - 46)

Spec. for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 - 46)

Method of Test for Expansion (Pin Test) of Copper and Copper-Alloy Tubing (B 153 - 45)

Spec. for Copper-Alloy Condenser Tube Plates (B 171 - 46)

Attention was called to the above noted corrections to the B-5 report as preprinted, all of these corrections having been considered in the committee's letter ballot; and these changes were accepted, by unanimous consent, for inclusion in the action on the report and its recommendations.

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by J. R. Townsend, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Magnesium-Base Alloy Die Castings (B 94 - 44 T)

Spec. for Copper-Base (Brass) Alloy Die Castings (B 176 - 42 T)

Attention was called to the following correction to the B-6 report as preprinted: Under Activities of Subcommittees, the paragraph under Subcommittee IV has been revised to read as follows:

"Subcommittee IV offers the following tentative statement for further consideration by the membership and by Committee E-1 on Methods of Testing:

"In the tension testing of die cast aluminum, magnesium, zinc, and brass alloy specimens, the rate of stressing up to the yield strength shall not exceed 100,000 psi. per minute. Exceedingly slow rates of testing may result in creep and hence are to be avoided. Beyond the yield strength, the rate of straining shall not exceed 0.25 inch per inch per minute measured on the gage length of the specimen.

"Die-cast lead, and tin alloys are considered in a different category since they are not ordinarily tested for yield strength. The following tentative statement was suggested to cover these materials:

"'In the tension testing of die-cast lead and tin alloy specimens, the rate of strain shall be $\frac{1}{8}$ inch per inch per minute measured on the gage length of the specimen.'"

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by I. V. Williams, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Aluminum and Aluminum-Alloy Extruded Bars, Rods, and Shapes.

Accepted as Tentative, Revisions in:

Spec. for Magnesium-Base Alloy Sand Castings (B 80 - 45 T)

Spec. for Magnesium-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 93 – 45 T)

Spec. for Aluminum Sheet and Plate for Use in Pressure Vessels (B 178 – 46 T)

Spec. for Magnesium-Base Alloy Permanent Mold Castings (B 199 – 45 T)

Spec. for Aluminum and Aluminum Alloy Bars, Rods and Wire (B 211 – 46 T)

Withdrawal of Tentative:

Spec. for Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 – 46 T).

In presenting the report, the chairman presented a summary of the paper by F. M. Howell, "The Determination of Elongation of Sand-Cast Light Alloy Test Bars" which was appended to the report and had previously been presented at the March, 1947, meeting of Committee B-7.

Committee B-9 on Metal Powders and Metal Powder Products:

Report presented from manuscript by W. A. Reich, chairman, and the following action taken:

Accepted as Tentative:

Spec. for Sintered Metal Powder Structural Parts

Advisory Committee on Corrosion:

In presenting the report, C. S. Cole, secretary, called attention to the fact that this was the first time a report of the committee had been presented to the Society. The report outlined the history and present functions of the advisory committee, with particular reference to its responsibility for the acquisition and maintenance of test site facilities for atmospheric corrosion testing for all other committees of the Society. Appended to the report was a survey and history of A.S.T.M. corrosion testing from the beginning of the Society's activities in this field.

FIFTEENTH SESSION—WATER

THURSDAY, JUNE 19, 8 P.M.

SESSION CHAIRMAN: C. E. IMHOFF

Round Table Discussion on Identification of Water-Formed Deposits

Introduction—C. E. Imhoff, presented by the author.

Some Problems in Nomenclature in Mineralogy and Inorganic Chemistry—Michael Fleischer, presented by the author.

Use of the Spectroscope in the Determination of the Constituents of Boiler Scale and Related Compounds—Alton Gabriel, Howard Jaffe and Maurice Peterson, presented by Mr. Jaffe.

An Evaluation of the Test Methods for the Determination of Dissolved Oxygen in Deaerated Boiler Feed Water—J. F. Sebald, presented from manuscript by the author.

SIXTEENTH SESSION—FATIGUE OF METALS, EFFECT OF TEMPERATURE

FRIDAY, JUNE 20, 9:30 A.M.

SESSION CHAIRMAN: G. R. GOHN

Committee E-9 on Fatigue:

Report presented by R. E. Peterson, chairman.

Papers:

The following papers were presented:

Fatigue Characteristics of Some Copper Alloys—H. L. Burghoff and A. I. Blank, presented from manuscript by Mr. Blank.

The Fatigue Characteristics of Copper-Nickel-Zinc and Phosphor Bronze Strip in Bending Under Conditions of Unsymmetrical Loading—G. R. Gohn and W. C. Ellis, presented by Mr. Gohn.

Fatigue Characteristics of Rotating Beam *versus* Rectangular Cantilever Specimens of Steel and Aluminum Alloys—F. B. Fuller and T. T. Oberg, presented by title and summarized by Mr. Gohn.

The High-Temperature Fatigue Strength of Several Gas Turbine Alloys—N. L. Mochel and P. R. Toolin, presented from manuscript by Mr. Toolin.

Joint Research Committee on Effect of Temperature on the Properties of Metals:

Report presented from manuscript by N. L. Mochel, chairman.

Mr. Mochel then called upon E. A. Sticha to present from manuscript the report on Stability of Steels as Affected by Temperature, which is to appear as an Appendix to the report of the joint committee.

Papers:

The following papers were presented:

The Creep Characteristics of Copper and Some Copper Alloys at 300, 400, and 500 F.—H. L. Burghoff and A. I. Blank, presented from manuscript by Mr. Burghoff.

Erosive Effects of Gun Blast on Materials—Development of a Strong Brittle Alloy[15]—James A. Broadston, presented from manuscript by Mr. I. V. Williams.

Creep and Creep-Rupture Testing[15a]—G. V. Smith, W. G. Benz, and R. F. Miller, presented from manuscript by Mr. Robert H. Aborn.

SEVENTEENTH SESSION—MISCELLANEOUS MATERIALS

FRIDAY, JUNE 20, 9:30 A.M.

SESSION CHAIRMAN: PRESIDENT ARTHUR W. CARPENTER

Committee D-19 on Water for Industrial Uses:

Report presented by Max Hecht, chairman, and the following actions taken:

Accepted as Tentative:

Methods of Test for the Detection of Iron Bacteria in Industrial Waters

Method of Reporting Results of Chemical Analysis of Water-Formed Deposits

Recommended Practice for Application of X-ray Diffraction Methods to Water-Formed Deposits

Recommended Practice for Corrosion Tests in Industrial Waters (NDHA Method)

Accepted as Tentative, Revision in:

Method of Test for Manganese in Industrial Waters (D 858 - 45 T)

Methods of Test for Silica in Industrial Waters (D 859 - 45 T)

Methods of Sampling Plant or Confined Waters for Industrial Uses (D 510 - 41)

Adopted as Standard:

Recommended Practice for Sampling Boiler Water from Stationary Boilers (D 860 - 45 T)

Adopted as Standard, Revision in:

Method for Determination of the Hydroxide Ion in Industrial Waters (D 514 - 41)

A motion was passed, upon the recommendation of F. N. Speller, vice-chairman of Committee D-19 on Water for Industrial Uses, for a rising vote of appreciation in honor of Max Hecht, chairman, for the great service he has rendered to Committee D-19 and to the Society.

Committee D-5 on Coal and Coke:

Report presented by A. C. Fieldner, chairman, and the following actions taken:

Withdrawal of Standard:

Method of Sampling Coal for Analysis (D 21 - 40)

Committee D-6 on Paper and Paper Products:

Report presented by W. R. Willets, vice-chairman, and the following actions taken:

Accepted as Tentative:

Method of Test for Bleeding Resistance of Asphalted Papers at Elevated Temperatures

Method of Test for Copper Number of Paper and Paperboard, with the following editorial revisions:

Section 4 (*e*).—Add at end of paragraph "and standardize against sodium oxalate as primary standard."

Section 6(*d*).—Change second sentence to read "where a copper number greater than 6 is obtained, the result does not correctly indicate how much the value exceeds 6 and if the actual value is desired, re-run the test increasing the amount, etc."

Method of Test for Blocking Resistance of Paper and Paperboard, with an editorial revision in Section 3(*b*) to delete the metric equivalents

Method of Test for Determining Titanium Dioxide in Paper

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[15] Published in the ASTM BULLETIN, No. 148, October, 1947.

[15a] See p. 615.

Method of Test for Crease Retention of Wrapping Paper, with the following revisions:

Section 2(a).—Change "1 in. diameter" to "1 in. radius."

Sections 4(b) second line and 4(c), third line.—Change "1 in." to read "0.98 ± 0.02 in."

Adopted as Standard:

- Method of Test for Adhesiveness of Gummed Tape (D 773 – 43 T)
- Method of Test for Absorption by Bibulous Papers of Water and Writing Ink (D 824 – 45 T)
- Method of Test for Degree of Wet Curl of Paper (D 826 – 45 T)
- Method of Test for Edge Tearing Strength of Paper (D 827 – 45 T)

Accepted for Publication as Information Only:

- Method of Test for the Siliceous Grit Content of Paper and Paper Products
- Method of Test for Scuff Resistance of Paperboard, with the addition of a new Section 4 on Conditioning, containing a reference to A.S.T.M. Standard D 685 – 44

Committee D-10 on Shipping Containers:

Report presented, in the absence of the chairman, by L. C. Gilbert, and the following actions taken:

Adopted as Standard:

- Method of Drop Test for Shipping Containers (D 775 – 45 T)

Adopted as Standard, Revisions in:

- Method of Compression Test for Shipping Containers (D 642 – 45 T)
- Method of Test for Shipping Containers in Revolving Hexagonal Drum (D 782 – 46 T)
- Method of Incline Impact Test for Shipping Containers (D 880 – 46 T)

Committee D-11 on Rubber and Rubber-Like Materials:

Report presented by Simon Collier, chairman, and the following actions taken:

Accepted as Tentative:

- Methods of Test for Contact and Migration Stain of Vulcanized Rubber in Contact with Organic Finishes
- Method of Test for Plasticity and Recovery of Rubber and Rubber-Like Materials by Means of the Parallel Plate Plastometer
- Method of Test for Viscosity by Means of the Shearing Disk Viscometer

Accepted as Tentative, Revision in:

- Methods of Test for Compression Set of Vulcanized Rubber (D 395 – 46 T)
- Method of Test for Indentation of Rubber by Means of the Durometer (D 676 – 46 T)
- Method of Heat Aging of Vulcanized Natural or Synthetic Rubber by Test Tube Method (D 865 – 46 T)

Adopted as Standard:

- Spec. for GR-M Polychloroprene Sheath Compound for Electrical Insulated Cords and Cables Where Extreme Abrasion Resistance Is Not Required (D 753 – 44 T)
- Method of Test for Hydrogen Permeability of Rubber-Coated Fabrics (D 815 – 44 T)

Adopted as Standard, Revision in:

- Methods of Test for Abrasion Resistance of Rubber Compounds (D 394 – 46)

It was announced that the proposed revision of the Standard Method of Test for Adhesion of Vulcanized Rubber to Metal (D 429 – 39) in the form of a new tentative method would be presented to the Standards Committee subsequent to the Annual Meeting.

Committee D-12 on Soaps and Other Detergents:

Report presented by B. S. Van Zile, chairman, and the following actions taken:

Accepted as Tentative:

- Spec. for Sodium Bicarbonate
- Spec. for Borax
- Method of Total Immersion Corrosion Test of Water-Soluble Aluminum Cleaners

Accepted as Tentative, Revision in:

- Definitions of Terms Relating to Soaps and Other Detergents (D 459 – 46 T)

Withdrawal of Tentative Revisions of Standards:

- Spec. for Chip Soap (D 496 – 39)
- Spec. for Olive Oil Chip Soap (D 630 – 42)
- Spec. for Powdered Soap (Non-alkaline Soap Powder) (D 498 – 39)
- Spec. for Olive Oil Solid Soap (D 592 – 42)
- Spec. for Milled Toilet Soap (D 455 – 39), except as noted in Annual Report.
- Spec. for White Floating Toilet Soap (D 499 – 39), except as noted in Annual Report.

Committee D-13 on Textile Materials:

Report presented by H. J. Ball, chairman, and the following actions taken:

Accepted as Tentative, Revision in:

Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 - 46 T)
Methods of Testing and Tolerances for Glass Yarn (D 578 - 46 T)
Methods for Testing Wool Felt (D 461 - 45)

Adopted as Standard:

Recommended Practice for Universal System of Yarn Numbering (D 861 - 46 T)

Adopted as Standard, Revision in:

Method of Test for Hard Scoured Wool in Wool in the Grease (D 584 - 43)
Methods of Testing and Tolerances for Woven Glass Fabrics (D 579 - 44)
Methods of Testing and Tolerances for Woven Glass Tapes (D 580 - 44)
Methods of Test for Wool Felt (D 461 - 45)
Spec. for Textile Testing Machines (D 76 - 45)
Def. of Terms Relating to Textile Materials (D 123 - 46). several defs. in Part A and all of Part B from D 123 - 46 T

Sectional Committee on Fastness of Colored Textiles, A.S.A. Project: L14:

Report presented, in the absence of the chairman, by P. J. Smith.

Committee D-14 on Adhesives:

Report presented, in the absence of the chairman, by A. G. H. Dietz.

EIGHTEENTH SESSION—FERROUS METALS, PAINTS, AND MISCELLANEOUS SUBJECTS

FRIDAY, JUNE 20, 2.00 P.M.

SESSION CHAIRMAN: J. T. MACKENZIE

Committee A-1 on Steel:

Report presented by N. L. Mochel, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Tentative Minimum Requirements for the Deformations of Deformed Steel Bars for Concrete Reinforcement[16]
Spec. for High Tensile Strength Carbon-Manganese-Silicon Steel Plates for Boilers and Other Pressure Vessels[16]
Spec. for Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels[16]
Spec. for Manganese-Molybdenum Steel Plates for Boilers and Other Pressure Vessels[16]
Spec. for Steel Plates for Pressure Vessels for Service at Low Temperatures[16]
Spec. for Alloy-Steel Rounds Suitable for Oil Quenching to End Quench Hardenability Requirements
Spec. for Medium Carbon-Steel Bars to Mechanical Property Requirements[16]
Spec. for Hot-Rolled Strip of Structural Quality
Spec. for Steel Machine Bolts and Nuts and Tap Bolts[16]

Accepted as Tentative, Revisions in:

Spec. for Billet-Steel Bars for Concrete Reinforcement (A 15 - 39), comprising the following changes:[16]

Section 5.—In the table of chemical composition, change the maximum phosphorus content of acid bessemer steel from the present value of "0.10" to "0.11" per cent.

Table I.—Add a footnote letter *b* after "Deformed Bars" and add the following under the table as footnote *b*:

> Where deformations conforming to the Tentative Specifications for Minimum Requirements for the Deformations of Deformed Steel Bars for Concrete Reinforcement (A.S.T.M. Designation: A 305) are specified in the order or supplied by the producer, certain changes in elongation shall be accepted as follows:

	Structural Grade	Intermediate Grade	Hard Grade
Elongation in 8 in. min., per cent.....	$\frac{1\,200\,000^{a}}{\text{tens str}}$	$\frac{1\,100\,000^{a}}{\text{tens str}}$	$\frac{975\,000^{a}}{\text{tens str}}$
	but not less than 16 per cent[a]	but not less than 12 per cent[a]	....

Table II.—Add a footnote letter *a* after "Deformed Bars" and add the following under the table as footnote *a*:

> Where deformations conforming to the Tentative Specifications for Minimum Requirements for the De-

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[16] These recommendations, not preprinted, were accepted by unanimous consent, subject to favorable letter ballot of Committee A-1, which ballot has been favorable.

formations of Deformed Steel Bars for Concrete Reinforcement (A.S T.M. Designation: A 305) are specified in the order or supplied by the producer, certain changes in bend test requirements shall be accepted as follows:

Thickness or Diameter of Bar	Structural Grade	Intermediate Grade	Hard Grade
Under ¾ in.	180 deg. d = 2t	180 deg. d = 6t	90 deg. d = 6t
¾ in. or over	180 deg. d = 4t	90 deg. d = 6t	90 deg. d = 6t

Spec. for Rail-Steel Bars for Concrete Reinforcement (A 16 – 35), comprising the following changes:[16]

Section 4 (a).—Add a footnote letter *b* after the word "Deformed" in the heading for the last column in the table of tensile properties and add the following under the table as footnote *b*:

> Where deformations conforming to the Tentative Specifications for Minimum Requirements for the Deformations of Deformed Steel Bars for Concrete Reinforcement (A.S.T.M. Designation: A 305) are specified in the order or supplied by the producer, certain changes in elongation shall be accepted as follows:

	Deformed Bars
Elongation in 8 in., min., per cent	$\frac{975\,000^{a}}{\text{tens. str.}}$

Section 5 (a).—Add a footnote letter *a* after the word "Deformed" in the heading for the last column in the table of bend test requirements and add the following under the table as footnote *a*:

> Where deformations conforming to the Tentative Specifications for Minimum Requirements for the Deformations of Deformed Steel Bars for Concrete Reinforcement (A.S.T.M. Designation: A 305) are specified in the order or supplied by the producer, certain changes in bend test requirements shall be accepted as follows:

Thickness or Diameter of Bar	Deformed Bars
Under ¾ in.	90 deg. d = 6t
¾ in. or over	90 deg. d = 6t

Spec. for Boiler and Firebox Steel for Locomotives (A 30 – 46), comprising the following change:[16]

Section 13.—Revise Section 13 on marking to read as follows:

13 (*a*) The name or brand of the manufacturer, and the manufacturer's test identification, class and minimum of the range of the tensile strength specified in Section 6 for the grade ordered, and, in addition, customer's special identification serial number shall be legibly stamped on each finished plate in two places not less than 12 in. from the edges and on each butt strap near the center line not less than 12 in. from each end. Plates, the maximum lengthwise and crosswise dimensions of which do not exceed 48 in. shall have the marking stamped in one place approximately midway between the center and an edge. The manufacturer's test identification number shall be legibly stamped on each test specimen.

(*b*) Except when otherwise arranged with the manufacturer (see Paragraph (*e*)) plate shall be match-marked as defined in Paragraph (*c*), so that test specimens representing them may be identified. When more than one plate is sheared from a single slab or ingot, each plate shall be match-marked so that they all may be identified with the test specimen representing them.

(*c*) Each match-mark shall consist of two overlapping circles each not less than 1¼ in. in diameter, placed upon shear lines, and made by separate impressions of a single circle steel die.

(*d*) Match-marked coupons, except as provided for in Paragraph (*e*), shall match with the plates represented and only those which match mark properly shall be accepted.

(*e*) When arranged with the manufacturer (see Paragraph (*b*)), for plates rolled on mills with rotary side and automatic end shears, and for plates necessitating flame cutting because of thickness or circular plates, making match marking impracticable, the manufacturer will furnish an affidavit, in lieu of the match-marking requirement, stating that the test coupons are representative of the plates and patterns cut from the material as indicated. If the plan of furnishing an affidavit is not satisfactory, the manufacturer and the purchaser may mutually agree on some other method of test identification.

Spec. for Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 – 46 T), with an additional revision deleting all references to deoxidized acid-bessemer pipe in the specifications.

Spec. for Axle-Steel Bars for Concrete Reinforcement (A 160 – 39), comprising the following changes:

Tables I and II.—Revise as indicated above for Tables I and II in Specifications A 15 – 39.

Spec. for Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 46)[16]

Spec. for Light Gage Structural Quality Flat Hot-Rolled Carbon-Steel (0.2499 and 0.1874 in. to 0.0478 in. in Thickness) (A 245 – 47 T)

Spec. for Light Gage Structural Quality Flat Rolled Carbon Steel (0.0477 to 0.0225 in. in Thickness) (A 246 – 47 T)

Spec. for Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 280 – 46a T)

Spec. for Carbon-Steel and Alloy-Steel Forgings for Magnetic Retaining Rings for Turbine Generators (A 288 – 46 T)

Spec. for Nonmagnetic Coil Retaining Rings for Turbine Generators (A 289 – 46 T)

Spec. for Carbon-Steel and Alloy-Steel Forgings for Pinions for Main Reduction Gears (A 291 – 46 T)

Spec. for Carbon Steel and Alloy-Steel Forgings for Turbine Generator Rotors and Shafts (A 292 – 46 T)

Spec. for Carbon-Steel and Alloy-Steel Forgings for Turbine Rotors and Shafts (A 293 – 46 T)

Spec. for Carbon-Steel and Alloy-Steel Forgings for Turbine Bucket Wheels (A 294 – 46 T)

Adopted as Standard:

Spec. for Carbon-Steel Bolting Material (A 261 – 44 T)

Spec. for Carbon-Steel Seamless Drum Forgings (A 266 – 44 T)

Spec. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 44 T)

Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 44 T)

Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for the Dairy and Food Industry (A 270 – 44 T), as revised in the A-1 Abbreviated Report.

Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 46 T)

Methods of Magnetic Particle Testing and Inspection of Heavy Steel Forgings (A 275 – 44 T)

Adopted as Standard, Revisions in:

Spec. for Carbon-Steel Axles for Locomotives and Cars and Tenders (A 21 – 36)

Spec. for Welded and Seamless Steel Pipe (A 53 – 46), with the following additional changes:[16]

Section 2(a).—Revise to read as follows: "The steel for both welded and seamless pipe shall be made by one or more of the following processes: open-hearth, electric-furnace, or acid-bessemer, except that steel for Grade B bessemer pipe shall be killed steel made by the deoxidized acid bessemer process (Note), and electric-resistance-welded pipe ⅛ and ¼ in. in diameter shall be made from open-hearth steel. The steel for furnace-welded pipe shall be of soft weldable quality.

NOTE.—Deoxidized bessemer steel for seamless pipe is an acid bessemer steel which has had a considerable portion of dissolved oxygen removed as a gas by treatment in the vessel and further treated in the ladle to develop a silicon content of 0.10 to 0.30 per cent and with sufficient aluminum, or its equivalent, to insure practically no evolution of gases during solidification.

Spec. for Hot-Rolled Carbon-Steel Bars (A 107 – 46), with additional changes[16] providing for both merchant and special bar qualities, bringing check analysis and silicon ranges into line with current practice, adding tolerances for bar size shapes, and omitting the present appendix covering estimated tensile values and suggested applications.

Spec. for Cold-Finished Carbon-Steel Bars and Shafting (A 108 – 46), with additional changes[16] which further revise and bring silicon ranges and check analysis into line with current practice, revising the inspection and rejection clauses to agree with Specifications A 107, revising dimensional tolerances to agree with the latest revision of A.I.S.I. Manual 9, and omitting the present Appendix covering estimated tensile values and suggested applications.

Spec. for Black and Hot-Dipped Zinc Coated (Galvanized) Welded Seamless Steel Pipe for Ordinary Uses (A 120 – 46)

Spec. for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 46)

Spec. for Carbon-Silicon Steel Plates of Ordinary Tensile Ranges for Fusion Welded Boilers and Other Pressure Vessels (A 201 – 46)

Spec. for High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (Plates 4½ in. and Under in Thickness) (A 212 – 46)

Spec. for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 46)

Spec. for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High Pressure Service (A 226 – 44)

Spec. for Hard-Drawn Steel Spring Wire (A 227 – 41)

Spec. for Carbon-Steel Valve Spring Quality Wire (A 230 – 41)

Spec. for Chromium-Vanadium Steel Valve Spring Quality Wire (A 232 – 41)

Spec. for Carbon-Steel Forgings for Locomotives and Cars (A 236 – 46)

Spec. for Alloy-Steel Forgings for Locomotives and Cars (A 238 – 46)

Spec. for Welded Alloy-Steel Boiler and Superheater Tubes (A 249 – 46)

Spec. for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 46)

Spec. for Welded Alloyed Open-Hearth Iron Pipe (A 253 – 46)

Revisions in Boiler and Pressure Vessel Plate Specifications—Stress Relieving—Tolerances—

Surface Finish and Conditioning.—In all boiler and pressure vessel plate specifications, namely:

A 30 - 46
A 129 - 46
A 201 - 46
A 202 - 46
A 203 - 46
A 204 - 46
A 212 - 46
A 225 - 46
A 285 - 46

revise the clause on stress relieving to read as follows:

> When so specified on the purchase order with the mill, test specimens representing the plates shall be stress-relieved by gradually and uniformly heating them to a temperature between 1100 and 1200 F. (or a temperature range otherwise agreed upon between the manufacturer and the fabricator), holding at temperature for at least 1 hr. per inch of thickness and cooling in still atmosphere to a temperature not exceeding 600 F.

Also in all the specifications listed above revise the table on permissible overweights of plates ordered to thickness by changing the first line under column 1 (Specified Thickness, in.) from the present "$\frac{3}{16}$ to $\frac{1}{4}$, excl." to read "Up to $\frac{1}{4}$, excl." For this thickness and a width of "48 in. and under" add a permissible overweight of "7" per cent to the table. For the same thickness and a width of "132 to 144, incl." add a permissible overweight of "21" per cent to the table.

Revise the section on bend test procedure and the table of bend test requirements to eliminate the use of the pin.

In all the boiler and pressure vessel plate specifications listed above retitle the section on finish to read "Surface Finish and Conditioning" and revise to read as follows:

> (*a*) The finished plates shall be free from injurious defects and shall have a workmanlike finish.
>
> (*b*) Plates not intended for riveted construction (see Paragraph (*e*)) may be conditioned by the manufacturer for the removal of surface imperfections or depressions on either surface by grinding, provided the ground area is well flared and the grinding does not cause the thickness to be less than the permissible minimum.
>
> (*c*) On plates intended for riveted construction the conditioning privilege described in Paragraph (*b*) does not apply unless agreed upon between the manufacturer and the purchaser.

Committee A-2 on Wrought Iron:

Report presented, in the absence of the chairman, by R. J. Painter and the following actions taken:

Adopted as Standard, Revision in:

Spec. for Wrought Iron Plates (A 42 - 39)

Reaffirmation of Standards:

Spec. for Refined Iron Bars (A 41 - 36)
Spec. for Iron and Steel Chain (A 56 - 39)
Spec. for Wrought Iron Rolled or Forged Blooms and Forgings (A 73 - 39)
Spec. for Staybolt Wrought Iron, Solid (A 84 - 39)
Spec. for Common Iron Bars (A 85 - 39)
Spec. for Staybolt Wrought Iron, Hollow-Rolled (A 86 - 39)
Spec. for Wrought Iron Rivets and Rivet Rounds (A 152 - 39)
Spec. for Uncoated Wrought Iron Sheets (A 162 - 39)
Spec. for Zinc-Coated (Galvanized) Wrought Iron Sheets (A 163 - 39)
Spec. for Rolled Wrought Iron Shapes and Bars (A 207 - 39)
Definitions of Terms Relating to Wrought Iron (A 81 - 33)

Committee A-3 on Cast Iron:

Report presented by E. R. Young, vice-chairman, and the following actions taken:

Adopted as Standard:

Spec. for Automotive Gray Iron Castings (A 159 - 44 T)
Spec. for Lightweight and Thin-Sectioned Gray Iron Castings (A 190 - 44 T)
Def. of Terms Relating to Cast Iron (A 196 - 46 T)
Rec. Practice for Evaluating Microstructure of Graphite in Gray Iron (A 247 - 41 T)
Rec. Practice for Torsion Tests of Cast Iron (A 260 - 42 T), with minor editorial changes.

Committee A-5 on Corrosion of Iron and Steel:

Report presented by T. R. Galloway, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Long Terne Iron or Steel Sheets with the following additional changes.[17]

Table I, Note b.—In the last line, substitute "871.12" for "971.12."

Section 8 (*b*).—Substitute "2.25 ± 0.01 in. square or 2.54 ± 0.01 in." for "2.250 ± 0.005 in. square or 1.540 ± 0.005 in."

Table III.—In the left hand heading, delete

the word "Steel." In the heading of the fifth column, delete the word "Approximate."

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[17] These recommendations, not preprinted, were accepted by unanimous consent, subject to favorable letter ballot of Committee A-5, which ballot has been favorable.

Method for Weight and Composition of Coating on Long Terne Sheets by the Triple Spot Test, with the following changes:[17]

Section 2, first line, substitute: "2.25 ± 0.01 in. square or 2.54 ± 0.01 in." for "2.250 ± 0.005 in. square or 2.540 ± 0.005 in."

Section 4 (d).—Change to read: "(*d*) Mercuric Chloride Solution.—Dissolve 50 g. of $HgCl_2$ in distilled water and dilute to 1 liter."

Section 4 (f).—Change to read: "(*f*) Standard Ceric Sulfate Solution.—Prepare as described for Reagent No 3 in Rec. Practice for Apparatus and Reagents for Chemical Analysis of Metals (A.S.T.M. Designation: E 50)."

Section 4 (h).—Change to read: "(*h*) Standard Iodate Solution.—Prepare as described for Reagent No 12 in Rec. Practice E 50 - 46 T, except that the solution shall be made 0.05 *N*."

Add a new Section 4 (j) to read: "(*j*) Sodium Bicarbonate Solution (Saturated)"

Section 6 (b).—Change seventh sentence to read: "Add 10 ml. of $HgCl_2$ (50 g. per l.) and 400 ml. of water."

Accepted as Tentative, Revisions in:

Spec. for Zinc Coating (Hot-Dip) on Iron and Steel Hardware (A 153 - 42 T), with the following additional changes:[17]

Table I: In the first column, under Class B-2, substitute "over 8 in. in length" for "any length." Add the following Notes 1 and 2 to this table, renumbering the present note as Note 3:

NOTE 1.—In the case of long pieces such as anchor rods and similar articles over 5 ft. in length, the weight of coating and uniformity test shall be the average of the determinations made at each end and the middle of the article. In the case of composite pieces each part shall be tested separately as they may fall in different classifications.

NOTE 2.—The number of specimens to be tested per order shall be agreed upon, at the time of purchase, by the manufacturer and the purchaser.

Adopted as Standard, Revisions in:

Spec. for Zinc (Hot-Galvanized) Coatings on Structural Steel Shapes, Plates, and Bars, and Their Products (A 123 - 33), with the following additional changes[17]

Section 5 (a).—Change to read as follows:

5. (*a*) Necessary precautions to fabricate properly and prepare the material for galvanizing to prevent embrittlement are described in the Recommended Practice for Safeguarding Against Embrittlement of Hot-Galvanized Structural Steel Products and Procedure for Detecting Embrittlement (A.S.T.M. Designation: A 143). Material found by these tests to be embrittled shall be rejected.

Section 9.—Delete the last sentence which reads: "The coating shall not interfere with the intended use of the material," and substitute "Globules or heavy deposits of zinc which will interfere with the intended use of the material will not be permitted."

Last year the committee had approved the transfer to the jurisdiction of Committee B-8 on Electrodeposited Metallic Coatings all specifications for cadmium coatings, including specifically, jurisdiction over A 165 - 40 T. At that time the question of the transfer of jurisdiction over specifications for electrodeposited zinc coatings on steel had been deferred. The committee reported that at the meeting just held it had reached the decision to transfer the Specifications for Electrodeposited Coatings of Zinc on Steel (A 164 - 40 T) to Committee B-8 which, it was felt, is in a better position to handle it. Committee A-5, however, in approving this transfer, makes two reservations: A-5 desires to retain the jurisdiction over specifications for products which carry zinc coatings, and it also requests that B-8 submit to A-5 for approval any proposed changes in A 164 - 40 T.

Committee A-6 on Magnetic Properties:

Report presented by Thomas Spooner, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Flat-Rolled Electrical Steel

Test for Permeability of Paramagnetic Materials, with the following changes: In Section 4 (*a*) the first line should read: "The thickness and width" instead of: "The length and width;" and in Section 5, in the denominator of the formula, "H" should have an exponent "2."

Adopted as Standard:

Core Loss Test for Frequencies up to 2000 Cycles and Ductility Tests of Magnetic Materials (A 34 - 46 T)

Editorial Changes Accepted in:

Method of Testing Magnetic Materials (A 34 - 46)

Definitions Relating to Magnetic Testing (A 127 - 46)

Committee A-7 on Malleable-Iron Castings:

Report presented by W. A. Kennedy, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Spec. for Malleable-Iron Castings (A 47 – 33)[18]
Spec. for Cupola Malleable Iron (A 197 – 39)[18]

Accepted as Tentative, Revisions in:

Spec. for Pearlitic Malleable-Iron Castings (A 220 – 44 T)[18]

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:

Report presented by J. J. B. Rutherford and the following actions taken:

Accepted as Tentative:

Spec. for Chromium-Nickel Corrosion Resisting Steel Spring Wire[19]
Spec. for Corrosion-Resisting Steel Billets and Bars for Reforging[19]

Adopted as Standard:

Spec. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 44 T)[19]
Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 44 T)[19]
Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for the Dairy and Food Industry (A 270 – 44 T)[19] (as revised) and
Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 46 T)[19]

These above four recommendations were presented with the joint cooperation of Committee A-1 on Steel.

Withdrawal of Standards:

Spec. for Chromium Alloy Steel Castings (A 221 – 39)[19]
Spec. for Alloy Steel 20 per cent Chromium 9 per cent Nickel Alloy Steel Castings (A 198 – 39)[19]
Spec. for Chromium-Nickel Alloy-Steel Castings (A 222 – 39)[19]
Spec. for Nickel-Chromium Alloy-Steel Castings (A 223 – 39)[19]

These have now been replaced by two new tentative specifications for Corrosion Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 – 46 T), and Heat Resistant Iron-Chromium and Iron-Chromium-Nickel for General Application (A 297 – 46 T).

Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Report presented by W. T. Pearce, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Mercuric Oxide for Use in Anti-Fouling Paints
Spec. for Dry Cuprous Oxide for Use in Anti-Fouling Paints
Method of Evaluating Degree of Resistance of Traffic Paint to Chipping
Methods of Testing Ethylcellulose

Accepted as Tentative, Revisions in:

Methods of Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow) (D 444 – 46 T)
Test for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions (D 563 – 45 T)
Methods for Producing Films of Uniform Thickness of Paint, Varnish, Lacquer, and Related Products on Test Panels (D 823 – 45 T)
Methods of Analysis of Diatomaceous Silica Pigment (D 719 – 45)
Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 46)
Methods of Chemical Analysis of Dry Cuprous Oxide and Copper Pigments (D 283 – 45)

Adopted as Standard:

Spec. for Yellow Iron Oxide, Hydrated (D 768 – 46 T)
Test for Daylight 45 Degree, 0 Degree Apparent Reflectance of Paint Finishes (D 771 – 44 T), as revised
Method of Evaluating Degree of Resistance to Flaking (Scaling) of Exterior Paints of the Linseed-Oil Type (D 772 – 44 T)
Method of Evaluating Degree of Resistance of Traffic Paint to Abrasion, Erosion, or a Combination of Both, in Road Service Tests (D 821 – 45 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 46)
Spec. for Ocher (D 85 – 41)
Spec. for Perilla Oil, Raw or Refined (D 125 – 41)
Methods of Testing Oleoresinous Varnishes (D 154 – 43)

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[18] These recommendations, not preprinted, were accepted by unanimous consent, subject to favorable letter ballot of Committee A-7, which ballot has been favorable.

[19] These recommendations, not preprinted, were accepted by unanimous consent, subject to favorable letter ballot of Committee A-10, which ballot has been favorable.

Spec. for Lampblack (D 209 - 46)
Spec. for Bone Black (D 210 - 46)
Spec. for Chrome Yellow and Chrome Orange (D 211 - 43)
Spec. for Pure Chrome Green (D 212 - 46)
Spec. for Reduced Chrome Green (D 213 - 46)
Spec. for Iron Blue (D 261 - 46)
Spec. for Ultramarine Blue (D 262 - 46)
Spec. for Reduced Para Red (D 264 - 41)
Spec. for Zinc Dust (Metallic Zinc Powder) (D 520 - 41)
Methods of Testing Drying Oils (D 555 - 41)
Test for Light Sensitivity of Traffic Paint (D 712 - 46)
Methods of Testing Liquid Driers (D 564 - 43)
Methods of Chemical Analysis of White Pigments (D 34 - 39)
Spec. for C. P. Zinc Yellow (Zinc Chromate) (D 478 - 41)
Test for Consistency of Exterior House Paints and Enamel-Type Paints (D 562 - 44)

Withdrawal of Standards:

Spec. for Toxic Ingredients in Anti-Fouling Paints (Dry Cuprous Oxide, Dry Mercuric Oxide) (D 277 - 31)
Test for Reactivity of Paint Liquids (D 479 - 40)

The recommendation for publication as tentative of the Method of Test for Measurement of Dry Film Thickness of Paint, Varnish and Lacquer Products, which appeared in the preprinted report, was withdrawn by the committee.

Committee E-1 on Methods of Testing:

Report presented by J. R. Townsend, chairman-elect, and the following action taken:

Adopted as Standard:

Hardness Conversion Tables for Steel (Relationship Between Diamond Pyramid Hardness, Rockwell Hardness, and Brinell Hardness) (E 48 - 43 T)

Adopted as Standard, Revisions in:

Spec. for A.S.T.M. Thermometers (E 1 - 46),[20] comprising a revision in the Low Cloud and Pour Test Thermometers 6C and 6F in accordance with the revision in the Standard Method of Test for Cloud and Pour Points (D 97 - 39) recommended by Committee D-2, which changes the lowest point of the Centrigrade thermometer 6 C. from "-60 C." to read "-80 C." and in the Fahrenheit thermometer 6 F. from "-70 F." to read "-112 F."

Committee E-3 on Chemical Analysis of Metals:

Report presented, in the absence of the chairman, by R. J. Painter, and the following actions taken:

Adopted as Standard:

Methods of Chemical Analysis of Steel for Sulfur by Direct Combustion and for Nitrogen (E 30 - 46 T)
Methods of Chemical Analysis of Ferro-Alloys (E 31 - 46 T)

Committee E-4 on Metallography:

Report, not preprinted, presented, in the absence of the chairman, by R. J. Painter.

Committee E-11 on Quality Control of Materials:

Report presented, in the absence of the chairman, by R. J. Painter.

Joint Committee on Filler Metal:

Report presented, in the absence of the chairman, by R. J. Painter.

NINETEENTH SESSION—TECHNICAL PAPERS AND REPORTS ON PLASTICS AND WOOD

FRIDAY, JUNE 20, 2:00 P.M.

SESSION CHAIRMAN: ALBERT G. H. DIETZ

Committee D-7 on Wood:

Report presented, in the absence of the chairman, by J. A. Liska, and the following actions taken:

Accepted as Tentative, Revisions in:

Methods of Testing Small Clear Specimens of Timber (D 143 - 27)

Adopted as Standard:

Methods of Testing Plywood, Veneer, and Other Wood and Wood-Base Materials (D 805 - 45 T)

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[20] This recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee E-1, which ballot has been favorable.

Committee D-9 on Electrical Insulating Materials:

Report presented by W. A. Zinzow, secretary, and the following actions taken:

Accepted as Tentative:

Spec. for Nonrigid Polyvinyl Tubing
Method for Sampling Electrical Insulating Oils
Test for Power Factor and Dielectric Constant of Electrical Insulating Oil of Petroleum Origin

Accepted as Tentative, Revisions in:

Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 – 46 T)
Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 – 43 T)
Spec. for Flexible Treated Cotton and Rayon Sleeving Used in Electrical Insulation (D 372 – 45 T)
Spec. for Vulcanized Fiber Sheets, Rods, and Tubes Used for Electrical Insulation (D 710 – 43 T)
Test for Rockwell Hardness of Plastics and Electrical Insulating Materials (D 785 – 44 T), subject to concurrence of Committee D-20.
Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used in Electrical Insulation (D 902 – 46 T)
Methods of Testing Sheet and Plate Materials Used in Electrical Insulating Materials (D 229 – 46)

Editorial Change Accepted in:

Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (D 392 – 38)

It was announced that the proposed revisions in the Tentative Methods for Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 46 T), which appeared in the preprinted report, would, after further minor changes, be submitted to the Society through the Standards Committee for publication as tentative.

The editorial note recommended for inclusion in the Standard Methods of Test for Grading and Classification of Natural Mica (D 351 – 38) was withdrawn from the report by the committee.

Committee D-20 on Plastics:

Report presented from manuscript by A. J. Warner, secretary. It was announced that the committee would present to the Standards Committee, subsequent to the Annual Meeting, several new tentatives and revisions in existing tentatives.

Papers:

The following papers were presented:

Universal Plastics Testing Machine—G. S. Brown, G. S. Burr, W. J. Gailus, L. D. Lipshutz, J. O. Silvey, S. Yurenka, and A. G. H. Dietz, presented from manuscript by Mr. Dietz.
Hardness and Abrasion Resistance of Plastics—L. Boor, J. D. Ryan, M. E. Marks, and W. F. Bartoe, presented from manuscript by Mr. Boor.
Influence of Nonhomogeneity of Wood on Its Strength Properties[21]—E. George Stern, presented from manuscript by the author.
Investigation of the Resistance to Impact Loading of Plastics[22]—Halvard Liander, Cyrill Schaub, and Arthur Asplund, presented from manuscript by Mr. Dietz.

There being no further business, the Fiftieth Annual Meeting was adjourned *sine die* by President Arthur W. Carpenter.

July, 1947.

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[21] Published in the ASTM BULLETIN, No. 147, August, 1947.

[22] Published in the ASTM BULLETIN, No. 148, October, 1947.

OUR STAKE IN MATERIALS PROGRESS

ANNUAL ADDRESS BY THE PRESIDENT

ARTHUR W. CARPENTER[1]

June 18, 1947

IN CELEBRATING, this week, the Fiftieth Annual Meeting of our Society, it is fitting that we should pause a moment and consider the origin of our great organization, some of its outstanding accomplishments in promoting knowledge of the properties and methods of test of materials and its future prospect so far as we may be privileged to see. Perhaps, then, we can appraise our stake in materials progress. The dictionary tells us that a stake is "an interest; something to be gained or lost." I am sure we should all agree that our many years of growth and productivity amply demonstrate the magnitude and worth of our interest in the field of materials. So far, the scale is tipped strongly on the side of gain. The only uncertainty in the appraisal is that of the extent to which we shall in the future increase that stake, and avoid the loss which must surely follow if we fail in any way to live up to our responsibilities. The satisfactory resolution of this uncertainty is strictly our concern and the concern of those who shall follow us in the carrying on of the activities of the A.S.T.M.

ORIGIN AND EARLY HISTORY

First of all, I think we are interested in knowing how this happens to be our Fiftieth Annual Meeting when we as a Society are not yet fifty years old. Organized interest in the properties and testing of materials goes back even farther than that—to the formation at Munich in 1882 of the International Association for Testing Materials, with the objectives: "The development and unification of standard methods of testing; the investigation of the technically important properties of the materials of construction and other materials of technical importance, and also, the perfecting of apparatus used for that purpose."

Several congresses were sponsored by this International Association and by 1898 the Association had grown to 1526 members from 22 countries, with 106 members from the United States. It was in that year, 1898, that the American Section was formed at a meeting of 20 members at the Engineers Club in Philadelphia. This group promptly arranged for the First Annual Meeting, held at the Philadelphia Engineers Club on August 27, 1898, consisting of an afternoon and an evening session, attended by 30 of its 70 members.

I wish I might tell you of each of the meetings that followed, for each was of interest. Suffice it to say that these were expanded to cover two or three days with some including plant visitations. From the very start thoroughgoing discussions were held on some important problems of testing or some properties of materials on which more information was sought. In the first year a committee was appointed, consisting of Professor Hatt and Edgar Marburg, which later presented a report on the subject "The Present State of Knowledge Concerning Impact Tests." This comprehensive and thorough report was really the first technical paper published by the Society. But there were no simultaneous sessions at these early meetings. Not until the early 1920's did we catch up with that 3-ring-circus idea.

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[1] Manager of Testing Labs., The B. F. Goodrich Co., Akron, Ohio

It was in the early years, too, that the forerunners of some of our most important committees were established. Those on steel, cement, paint, petroleum products, coal and coke, and wood were all active by 1904 or earlier and they continue now to function effectively.

SOCIETY INCORPORATED—LEARNED BY DOING

It was not long until the leaders at that period realized the need for a better type of organization than was possible in the American Section of the International Society—an organization which could function with more autonomy, provide protection with respect to individual liability, and permit more freedom of action in the matter of establishment of standards, particularly specifications, that would be useful in this country. In consequence, the American Society for Testing Materials was legally incorporated through the granting of a Charter on June 3, 1902, under the laws of Pennsylvania, and this Charter was unanimously adopted by the membership at the fifth Annual Meeting held June 12 to 14 here at Atlantic City. We might have wished for a more expressive title for A.S.T.M. but we take no issue with the statement of purposes; they are sufficiently comprehensive to include all activities in development and standardization of specifications, of methods and equipment for testing, as well as research investigations and publication of reports and papers dealing with the constitution, structure, and properties of engineering materials. Affiliation was maintained with the International Association until it ceased to function at the time of the first World War. With the granting of the Charter, a new organization was born, but the meetings it held were still of the same character as before and the numbering was continued. Therefore, while we are now celebrating our fiftieth Annual Meeting, we can still look forward to our real golden anniversary in 1952.

"The new Society had many obstacles to overcome"—here I can do no better than to quote Professor Arthur N. Talbot, himself an early President of the Society—"It was without precedents. It learned to do by doing. It developed methods, procedure, standards. It encouraged research and recognized its value wherever found. It fostered cooperation. It coordinated the efforts of the many. Soon it had expanded its field and trebled and quadrupled the number of its working committees. Before long it had established a reputation for fairness of procedure, correctness of information, soundness of judgment, fullness of research data, and the stability of its organization. Its specifications and standards became accepted in the engineering world. The Society began to take a high place among the organizations in the technical field and to supply a most useful purpose to the business world. Besides, its activities gave opportunities and development to a body of young and growing members, which in itself was a high service to the industrial and engineering world well worthy of recognition."

EARLY LEADERS

The Society was particularly fortunate in the selection of its first president and first secretary. Dr. Charles B. Dudley, chief chemist of the Pennsylvania Railroad, served as President from 1902 until 1909, and Dr. Edgar Marburg, Professor of Civil Engineering at the University of Pennsylvania, was Secretary-Treasurer from 1902 until 1918. These men of vision, courage, and organizing genius surrounded by a distinguished and talented group of pioneers in a new field laid the foundation and built the framework on which the Society has made such enormous progress. Among others who stand out in the group of charter signers, Professor Talbot mentioned Robert Lesley, attributing to him the valuable contribution of having poured oil on the sometimes troubled waters between groups of producers and consumers. To the name of Mr. Lesley, I would add that of Henry M. Howe. I was pleased to see the quotation from his annual address in 1902 that appeared in the May issue of the ASTM BULLETIN, for it shows the sort of thinking that went into the Society and its operations. We are indebted to these early leaders for establishing the sound fundamental principles underlying A.S.T.M. work. From the very start everyone who had an interest was to be given an opportunity to participate in its deliberations. The pattern of cooperation thus set has permeated all A.S.T.M. activities.

Looking back, we might wish that less emphasis had been placed in those early days on the prompt promulgation of materials specifications until test methods had been more thoroughly perfected. We all realize that reliable, reproducible methods of measurement of properties are necessary prerequisites for the preparation of satisfactory, workable specifications. Much argument and criticism might have been avoided and ultimate progress expedited if there could have been more attention given to the cultivation of better understanding and more thorough standardization of methods of testing. However, the pressure for specifications is readily understood when the dire needs of industry for uniform materials in mass production are appreciated. Perhaps, too, the emphasis on specifications was not without merit in pointing up the need for exact test procedures, and it certainly was instrumental in promoting numerical growth and broad representation. It has been said that many producers first participated in the work of the Society to keep from being hurt by specifications formulated by consumers. These producers soon were persuaded of the advantages of wholehearted cooperation and remained as most interested and industrious workers.

Dr. Marburg was succeeded as

Secretary-Treasurer in 1918 by our own Mr. Warwick, who had been associated with him over a number of years. I think that the Society is fortunate in having had, through Mr. Warwick and his staff, such intimate contact with those who were responsible for establishing its principles of operation. Several of the present staff knew a number of these men personally. They are familiar with the procedures that have been set up and the reasons for them. With their help, we shall continue to meet successfully one of our first responsibilities, that of reexamining from time to time the procedures established earlier and making modifications to keep them in tune with changing conditions. As our technical committees grow in size, new methods of operation must be devised—but we must continue to adhere to our basic principles of full representation and voluntary cooperation.

WORK ON STANDARDS

The first dozen specifications which had been prepared and approved by the American Society, for the most part related to iron and steel. Incidentally, none of these standards covered methods of testing other than those included in the materials specifications, apart from inferred dependence on current practices. The number of specifications has continued to grow, and of late at a greater rate—there were 28 in 1910, 110 in 1915, double that number in 1920, and by 1930 we were well over the 500 mark. But in the period from 1940 to 1946 we completed almost another 500. In short, it took us about 30 years for the first 500, 10 years for the next 500, but only about 6 years for the most recent 500—a rapidly accelerating rate indicating true zeal and real worth in the activities in our committees. Of the present number of standards and tentatives, over 1400, more than half, are for test methods and definitions. Altogether, they deal with several hundred different materials and require nearly 7000 pages in five volumes for their publication. However, the mere number is less important than the acceptance that these standards have been accorded. They are written into building and other types of codes throughout the country. Many of them form the basis of business transactions representing a considerable bulk of the manufactured product of the nation. They find wide use in control of our industrial production and process efficiency.

Here again, a very great responsibility is involved for maintaining the quality of these standards. I am sure our technical committees and the Administrative Committee on Standards keep this constantly in mind. We can depend on the able chairman of the latter, Mr. R. D. Bonney, to pursue this matter thoroughly. But it is not a responsibility of the committees alone. Every single member must be jealous of the quality of A.S.T.M. standards and see that any new addition that is made to the present number fully merits approval as such. The present ones need constant review to eliminate those that have outgrown their usefulness and to see that they keep abreast of developments in materials. New ones must be established where desirable and should be ready when needed. It is not enough to wait for demand.

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We should lead. For this, we must ever be alert to the advancing front of knowledge and to the development of new methods and techniques as well as to the introduction of new materials. For example, where application of statistical quality control methods will aid in production and assure greater uniformity, thereby helping both producer and consumer of a particular product, we should be working now to broaden our knowledge of these applications and to adapt our materials specifications to such programs. And again I would single out methods of testing. Our committees will need to give more and more attention to the question of the significance of tests and the evaluation of our test procedures. One of our most important standards today covers the verification of testing machines—testing testing machines if you will. Likewise, we need better means for the verification of our test methods. What do they show? How reliable are they?

MEMBERSHIP—YOUNG GROUP

At the Annual Meeting in 1902 which approved the Charter, the membership was reported as 175, having more than doubled the original 70 of the American Section. During the following year this increased to 349. The members, then as now, worked in technical committees, of which five reported at the next annual meeting. Today, our membership is more than 6200, comprising individuals, companies, universities, government agencies, associations, and the like, from among whom nearly 4000 individuals are actively at work in some 70 technical committees on research and standardization problems. Their reports and standards command respect throughout the world. May I remind you that at this annual meeting alone we are having more than 350 meetings of our technical committees? We are particularly proud of our growth in personnel, for it indicates the very great interest in our stake in materials. We are responsible for seeing that this interest is maintained, as we must always have an active body of working technical men. This is our greatest asset. You will recall that Dr. Talbot, in the address quoted earlier, referred to the fact that the "activities (of the new Society) gave opportunities and development to a body of young members." That is important and must be maintained. We think of the founders as "Grand Old Men" and so they were—but in their day, they were far from being old, either in years or in ideas. Dr. Marburg was in his thirties when elected Secretary-Treasurer. We must do all we can to enlist the support of the young technologist in all our activities and particularly in our committee work.

SOME ACCOMPLISHMENTS

In reviewing the history of our founding and a few of the outstanding features of our growth, it is obvious that reference to many important activities of our Society has necessarily been omitted. It would be impossible within reasonable time limitations to begin to cover adequately the many phases of an organization which operates in a cross-section of all industry and is concerned with all sorts of technical activities. The magnitude of our $400,000 annual budget alone gives some idea of the extent of our operations. Mention might have been made of our District activities with eleven Councils established and holding meetings regularly in important industrial centers from coast to coast. Then, there are numerous publications in addition to our regular Book of Standards and *Proceedings*—the compilations of standards for individual industries, special books and pamphlets covering symposia and other presentations of technical information in selected fields, joint publications with other technical societies and our BULLETIN which is rapidly approaching the status of a technical journal. These all help in our work of promoting and disseminating knowledge of the materials of engineering. Particularly important are certain of the research projects, some of which were initiated early in the history of the Society and which are continuing to aid greatly in the development of materials for special services. Among these may be mentioned the Society's work on Corrosion and Fatigue of Metals and the joint project with A.S.M.E. on the properties of metals at high and low temperatures. We need not dwell on our accomplishments; they are so generally accepted. Enough has already been said to establish without any doubt that our stake in materials progress has grown very large with the years and has brought with it grave responsibilities for the future.

The two World Wars were times when the value of Society work became particularly evident. Not only were its standards of inestimable worth, particularly in the last war in aiding procurement, but its committees and its personnel were called upon in large measure to aid in solving the many materials problems. In each instance the two wars emphasized the need for more information. At the time of the first war we would have liked to know the effects of increased phosphorus and sulfur limits in steel. Immediately following the war, investigations were undertaken to solve this moot question. The recent war pointed out the need for a great deal more information on replacement materials in view of the many shortages that developed. Adequate data on the properties of some of the newer materials would have been of tremendous value. Data were lacking on the behavior of metals and other materials at high temperatures for use in gas turbine and jet propulsion engines. Many exposure conditions were encountered that required special treatment. There was need for protection methods and better packaging and handling procedures, to mention but a few.

EMPHASIS ON RESEARCH

There is still much work to be done on standardization—utilizing and applying in new ways the vast store of technical information which has been accumulated. But the real opportunity in the future will, I believe, depend on extending our knowledge through much expanded emphasis on research. Great changes have taken place in the last decade (yes, even in the past two or three years) which will bring us face to face with materials problems so gigantic and perplexing that we cannot hope to meet them unless we delve more deeply in search of knowledge now unknown.

One outstanding way in which the necessary information is developed is by holding special symposia or round-table discussions, of which there are so many on the program for this Annual Meeting. These are all on the initiative of some committee or group and took no urging. For every one scheduled, probably many more could be held, limited only by the number of days available and the means for publication. There are almost as many already scheduled for the 1948 meeting in Detroit. In addition, many committees are sponsoring their own independent discussion sessions and at least one committee is thinking of arranging a lecture series. All of this indicates another big responsibility, that of publishing and disseminating the data. For while some of the discussions have served their purpose through accomplishing an exchange of ideas, many should reach publication to secure their full value.

We have prospered greatly in the past and are rich in material things far beyond the dreams of the early pioneers of our Society. We have freely used the technical information obtained by industry and brought to us by our members. We have asked them to undertake assigned research tasks and give us the results which they have generously and willingly done. I believe that all means of getting needed information should be fostered—the private institution, the individual research worker, organized committee effort and that being carried out by public institutions. And we should find the means of making this information available. I pass that along as a challenge to our good friends on the Administrative Committee on Research to attempt to work out some means for mobilizing all research efforts to bear on problems the committees have in hand.

RESEARCH FUND

Organized research, too, has its place. On special problems of particular interest to certain specific industries, we have gone to them and obtained substantial contributions of funds to finance the necessary research. In the case of corrosion, this has amounted over the years to more than $100,000, aside from contributions of materials. Similar smaller funds provided for the work on fatigue and the effect of temperature on metals, and other problems. Seldom, however, have our committees sought to determine through broad surveys what technical information might be useful and then initiated projects for obtaining it unless such projects were strongly supported by industry. Likewise, standardization has generally depended on prior accumulation by industry of the required knowledge. These methods of operation have not reflected lack of initiative but, rather, the taking in hand of immediate problems with no thought as to what could be done on a broad basis if the funds were available.

This situation has been repeatedly discussed in Presidential Addresses of the past. In 1931, Mr. K. G. Mackenzie called attention to the Research Fund of $8387.50, the income from which would not cover a single problem with one worker at some university or technical school. He then said, "There is work to be done; we must do it." Again in 1932, Dr. Clements pointed out that it behooves our Society to pin its faith to the continual search for and dissemination of new knowledge. He suggested that we greatly augment the principal of the Research Fund, and set a goal of $500,000. He further suggested private gifts of stock or money, the return to come to the donor for life and the principal at death to go to the A.S.T.M. Research Fund. He later gave point to the suggestion by setting the first example. The fund was again discussed in 1934 by Professor Lawson, but the country was still in the depths of depression and no tangible progress could then be made. Through gifts and resumption of the policy of adding to the fund from entrance fees, the principal has now grown to about $45,000, which is substantial but far short of that needed to provide sufficient income to finance research on any projects of general interest in the field of materials to be carried out, perhaps, under fellowships in universities or technical schools.

I am not suggesting that I think, by spotting research fellowships all over the country, we can just turn on a faucet, and get the answer to any problem. But frequently, I feel sure, there are investigations that could be greatly expedited if some research man could be assigned to a key problem. Sometimes a very little help would do much to finish a job already partly completed. Often a project needs a bibliography, and while we have been fortunate in this respect in finding public-spirited cooperators, sometimes these individuals are not available. In some cases, the need is only a question of publication. Consider also a rather general investigation, as, for example, a study of one of the new composite materials. Can we expect the supplier of an individual component, of which perhaps only a small amount is used, to underwrite the investigation? Or can we depend on the potential user, faced with reaching a decision between this or a number of other products, to do all of the research work? Sometimes knowledge secured just for the sake of completing our knowledge may be worth while. Who knows?

There are many basic problems still unanswered. Among specific subjects which need research may be mentioned the property of hardness and its measurement, the fundamental nature of adhesion, the mechanism and measurement of abrasive wear, and there are many others. Their solution should not wait for the slow accumulation of funds from entrance fees nor for the death of some generous benefactor. We shall lose some of our stake in materials progress unless we find a way to continue to render the service which is expected of us.

Now that we have completed our new headquarters building and shall doubtless soon solve our other financial problems incident to operating under the changed economic conditions which face us, I should like to propose that we again take up the matter of providing more adequately for research. Dr. Clements' goal is none too large, and if we really go to work on it, we will not fail. What a splendid accomplishment it would be if we could celebrate our golden anniversary in 1952 with a Research Fund in keeping with the distinguished record of our past. Thus, we would in some measure assure the preservation of our stake in materials progress.

AHEAD

The future holds so much in store, with the many new materials that should be explored, and the new methods needed to meet the many and diverse conditions that are being encountered. Low temperatures, high temperatures, humidity, enormous stresses, chemical corrosion—and soon, Dr. Marshall, our speaker of the evening, will no doubt present many more. We have recently organized a new Administrative Committee on Simulated Service Testing recognizing that frequently materials cannot be completely evaluated from a laboratory specimen of some restricted form but must be tested under actual use conditions. Apart from this, performance testing in general has always presented an inviting field.

With all these new types of test, new materials to be tested, new conditions to be met, it behooves us to be prepared.

As Tennyson wrote many years ago:

> "Yet I doubt not thro' the ages one
> increasing purpose runs,
> And the thoughts of men are widen'd
> with the process of the suns.
> Knowledge comes, but wisdom
> lingers, and I linger on the shore,
> And the individual withers, and the
> world is more and more."

ANNUAL REPORT OF THE BOARD OF DIRECTORS*

This first report of the new Board of Directors to the Society covers a year of much work both in the Board and throughout the Society, carried on under conditions of considerable economic and business uncertainty. "Reconversion" of our technical activities from war to a peace-time basis was accomplished smoothly and effectively over a year ago, and throughout the committees of the Society, with very few exceptions, there is in evidence a strong spirit of accomplishing things and of working to advance our knowledge of materials and to produce for the use of our industries and technologists adequate tests and specifications for the materials of engineering.

The Board in this report, together with the reports of the Administrative Committees appended, has endeavored to present this picture in outline; obviously the whole picture can be seen only in the wealth of material presented to the members at this annual meeting and in the many publications that have been issued and are being planned. One must look, too, at the record of many fine meetings held during the year in the eleven districts of the Society, which are merely listed in the report but which carry the meaning of the Society and its work to our members and others throughout the country.

The steady growth of membership is inspiring, and so, too, is the bringing of new members into our committee work which goes on continuously and especially so, of course, in those fields of work in which new committees are being organized.

The Board of Directors has given much thought this year to the problems involved in the program of expansion and development of technical activities announced two years ago and covered quite fully in the 1946 report. The incidence upon this program of the rapidly rising costs which for well over a year have been serious and show no signs of abating, has finally led the Board to the conclusion that our activities can no longer be supported at the present level of membership dues and that an increase in these dues is essential to the future development of the Society upon sound and substantial lines. Accordingly, in the section of this report dealing with Finances, the Board is recommending an amendment of the By-laws to effect such an increase in dues and presents there the financial status of the Society and the considerations that have led to this recommendation.

TECHNICAL ACTIVITIES

The technical work of the Society has been actively carried on during the year, as will be seen in the committee reports for this meeting and the number of new specifications and methods submitted for action. The Administrative Committee on Standards is responsible for following the standardization activities of the Society and its annual report to the Board (see Appendix II) records a number of interesting developments. The Administrative Committee is giving particular attention to improving the quality of A.S.T.M. standards from editorial and technical points of view. It is worthy of note, too, that several of the technical committees are engaged in extensive reorganization to provide for more efficient handling of expanded activities. Attention is also being given to the adequacy and representativeness of the personnel of some of the technical committees, particularly with respect to the consumer interests.

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*Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

The Administrative Committee on Research, whose annual report to the Board appears as Appendix III, has reviewed the research activities being carried on by the technical committees and has published a summary of such projects in the May BULLETIN. It is gratifying to note that many new projects have been started during the past three years since the previous summary was published. The Research Committee has begun a series of conferences with technical committee officers on research projects, looking to stimulation of such activities in the committees and the setting up of suitable committee organization. The committee has also written to all committees in respect to applications for grants from the A.S.T.M. Research Fund.

Two other administrative committees deal with phases of our technical activities, namely, Administrative Committee on Ultimate Consumer Goods and Administrative Committee on Simulated Service Testing. The annual reports of these two committees appear as Appendices V and VI, respectively, and record the progress made in the fields of work assigned to them. In the field of consumer goods a study is under way of the various factors entering into the development of test methods for such goods, with respect both to intrinsic qualities and to those characteristics that make a thing "wantable" to the consumer. In the field of simulated service testing, work on such testing has already begun in certain committees and the Administrative Committee is studying the development of this type of work for bearings, springs, and sandwich constructions.

The plan of assigning staff engineers to keep in touch throughout the year with the activities of the technical committees, as described in previous reports, has been further developed during the year. There are now five members of the staff who regularly follow the activities of certain technical committees, working under the direction of the Technical Secretary. They attend meetings of the committees and many of the subcommittees, and particularly meetings of the advisory committees. It is believed that the services they are able to render aid in smoother and more effective conduct of our committee work and are much appreciated by the committees.

New Activities:

Two new technical committees have been organized, both in May, 1947; Committee C-2 on Oxychloride Cement, under the chairmanship of L. S. Wells, National Bureau of Standards, and Committee D-15 on Engine Anti-freezes, under the chairmanship of H. R. Wolf, General Motors Corporation. The personnel and scopes of these two new committees will appear in the next edition of the Year Book.

In the report a year ago reference was made to the growing importance of so-called "Sandwich-Type Constructions" and of the need for development and standardization of adequate tests and specifications to cover these composite materials. A conference on June 7, 1946, of committee representatives and of producers and consumers of these types of constructions and materials served to define the problem in this field and indicated the need of further study. Accordingly the Board of Directors appointed a special committee to give further consideration to the subject and to submit specific recommendations. This committee, consisting of L. J. Markwardt, chairman, R. B. Crepps, J. M. Frankland, G. M. Kline, R. C. Platow, G. M. Rapp, and R. L. Templin, has presented a comprehensive report to the Board, which concluded with the following four recommendations:

1. That consideration of sandwich construction problems by the Society be limited for the present to "structural sandwich" constructions;
2. That a committee on "structural sandwich constructions" be established within the Society as soon as practicable, to be responsible for problems relating to structural sandwich constructions. So far as possible this committee should call on existing technical committees for test procedures and specifications relating to individual component materials. When the need arises for test methods and specifications on a component material not within the scope of existing committees, the new committee should proceed independently to meet its own problem with respect to its own field of work;
3. That the Committee on Structural Sandwich Constructions develop such test methods and specifications for sandwich constructions as may be necessary, including not only methods for mechanical tests, but also methods for other tests such as durability, using the existing facilities of other technical committees as far as possible; and
4. That the Committee on Structural Sandwich Constructions give consideration to the needs for simulated service tests and tests of end products in this field.

Based on these recommendations the Board of Directors has authorized the formation of a Technical Committee on Structural Sandwich Constructions. The special committee has been continued to advise with the President and Executive Secretary in the selection of personnel, determination of scope, and relationship to be established between the new committee and the present committees of the Society dealing with the various constituent materials of sandwich constructions, such as metal, wood, rubber, plastics, adhesives, textiles, glass, and paper.

Consideration during the year of a proposal to organize a new technical committee on Appearance Standards, as referred to in the report of a year ago, developed support from a number of the committees of the Society. However, it has seemed desirable to give further consideration to work on this type of standards and arrangements are being made for a conference of representatives of interested committees and other members of the Society at which it is hoped that specific recommendations will be developed. M. Rea Paul has been designated to serve as chairman of this conference.

Developments during the war have emphasized the great importance of both research and formulation of test methods with respect to materials used in the construction of gas turbines and of jet propulsion engines where extremely high temperatures are involved. A Symposium on Gas Turbine Materials at the 1946 Annual Meeting, as well as a meeting on the same subject sponsored by the Philadelphia District Council, evidenced a very keen and widespread interest and led to a conference to consider the most effective means for the Society to work in this field. The conference suggested that the ASTM-ASME Joint Research Committee on Effect of Temperatures on the Properties of Metals, with suitable expansion of its scope and personnel, would be a logical group to undertake work in this field. At a recent meeting the Joint Committee agreed to such an expansion of its functions and is now engaged in the formulation of specific plans.

Reorganization of Committee E-1:

As noted in the report a year ago, the reorganization of Committee E-1 on Methods of Testing has been under consideration for some time, the committee work of the Society having developed so greatly and along so many lines since Committee E-1 was first organized on its present basis that a re-appraisal of the Society's procedure in the development and coordination of methods of test was advisable. During the year the Board's Committee on Technical Committee Activities, with the Advisory Committee of Committee E-1, has thoroughly reviewed the responsibilities of Committee E-1 and its relation to the technical committees in the development and coordination of A.S.T.M. methods of testing. The Board of Directors has reached the following conclusions:

1. The responsibility for the preparation of methods of testing and the examination, design, and recommendation of instruments, appliances and machines for testing will continue to be vested in the technical committees as now provided in Section 1 of the Regulations Governing Methods and Instruments of Testing (see Year Book, pp. 485–487).

However, the committees shall refer to Committee E-1 for its consideration and advice any proposed methods of testing, or specifications containing requirements for apparatus, or amendments of existing standards, preferably while in draft form and before presentation to the Society, but in any case prior to their reference to the Society for adoption as standard. (This provision is a substitute for Section 7 (b) of the above-mentioned Regulations.)

2. The general functions of Committee E-1 will continue to be those set forth in Section 6 of the above-mentioned Regulations.

3. The membership structure of Committee E-1 will be changed to consist of twelve members of the Society appointed by the Board of Directors having the qualifications now specified for the present "Members-at-large." The "Representative Members" now serving on the main committee as representatives of the technical committees will be discontinued, although representatives of all technical committees interested in any problems being studied by Committee E-1 will continue to serve on the working committees of E-1. (This provision will involve amendment of Section 4 of the above-mentioned Regulations as well as deletion of reference to "representative members" in Sections 5 and 7 a.)

4. Committee E-1 is to establish advisory groups on particular subjects, letting each such group appoint whatever task or working committees are necessary and making the advisory group responsible for seeing that such task or working committees are properly constituted and representative of the interested technical committees.

5. It shall further be the obligation of the technical committees to state in their annual reports whether any proposed method of testing, or specification containing requirements for apparatus, or amendments of existing standards, being recommended in the report have been submitted to Committee E-1 for review and also indicate what action has been taken by Committee E-1. (This provision is to be added to Sections 19 and 31 of the Regulations Governing Technical Committees.)

Such changes in the Regulations as are required to embody these provisions have been made by the Board after consultation with the chairmen of the technical committees and will appear in the 1947 Year Book.

There still remain to be considered some questions concerning the scope of Committee E-1, particularly in relation to the broad coordinating function of such committees as the Advisory Committee on Corrosion and the activities of such technical committees in the E group of committees that deal with specific fields of testing. These will be discussed with the new Committee E-1, which will assume its responsibilities following the 1947 Annual Meeting.

Committee Scopes and Jurisdiction:

Consideration has been given to an extension of the scope of the Advisory Committee on Corrosion whereby its responsibilities for coordination of corrosion investigations, researches, and test procedures within the Society would be expanded to include similar coordinating functions with respect to exposure testing of all materials. However, it was finally concluded that this would involve so diverse a field and such an expansion of personnel as seriously to affect the efficiency of operation of the committee in its present field, and the scope limiting the functions to the field of corrosion was confirmed.

The Regulations Governing Corrosion Testing have been embodied in the general Regulations Governing Technical Committees (see 1946 Year Book, pp. 488–490), which regulations include a statement of functions of the Advisory Committee and of the obligations of technical committees to cooperate with the Advisory Committee in all matters concerned with researches, tests, and specifications dealing with corrosion of metals. They also establish the responsibility of the Advisory Committee in the acquisition and maintenance of corrosion test sites, and provide for the time being that the Advisory Committee will also be responsible for the acquisition and supervision of test sites for the exposure testing of any material by a committee of the Society. If the use of such test sites for non-metallic materials should become quite extensive, some change in this arrangement may need to be considered.

Subcommittee IV, of Committee D-11, on Protecting Persons from Electrical Shock has been reorganized as a sectional committee under ASA procedure with the Society as administrative sponsor and the Edison Electric Institute as co-sponsor. This committee will assume responsibility for existing A.S.T.M. standards in this field as well as for war standards for linemen's protective equipment that were developed under emergency ASA procedure.

The scope of Committee D-2 on Petroleum Products and Lubricants has been expanded to assign to that committee the field of synthetic lubricants. The scope of Committee D-20 on Plastics has been extended to include the coverage of plasticizers and other raw materials used in the manufacture of plastics. The scopes of Committee B-1 on Wires for Electrical Conductors and Committee C-17 on Asbestos-Cement Products were revised to clarify and define the fields of those committees more accurately. Revised statements of scope were published in the 1946 Year Book..

The scope of Committee A-6 on Magnetic Properties has been extended to cover jurisdiction over the development of standards for properties of engineering materials associated with their magnetic properties, and the revised scope will appear in the 1947 Year Book.

During the year, jurisdiction for the Tentative Specifications for Electrodeposited Coatings of Cadmium on Steel (A 165) were transferred from Committee A-5 on Corrosion of Iron and Steel to Committee B-8 on Electrodeposited Metallic Coatings, with the understanding that the latter committee would hereafter be responsible for specifications covering electrodeposited coatings of cadmium on steel. Appropriate modification of the scope of Committee B-8 was made in the 1946 Year Book.

Minor changes in scopes of Committee A-7 on Malleable-Iron Castings and Committee C-1 on Cement have been approved; the revised scope of the latter committee was published in the 1946

A.S.T.M. MEETINGS JUNE, 1946–MAY, 1947, INCLUSIVE

Meeting	Place and Date	Notes
Annual Meeting	Buffalo, N. Y. Hotel Statler June 24–28, 1946	25 Technical Sessions 200 Committee Meetings 2 Exhibits (1835 in attendance)
A.S.T.M. Committee Week	Philadelphia, Pa. Hotel Benjamin Franklin February 24–28, 1947	250 Committee Meetings. (1260 in attendance)
Spring Meeting	Philadelphia, Pa. Hotel Benjamin Franklin February 25, 1947	Symposium on Paint and Paint Materials (two sessions) (About 500 in attendance at each session)
District or Local (sponsored by Councils Indicated)	San Francisco, Calif. (Northern California) Moffett Field September 27, 1946	Tour of Field, including Laboratory Discussion Period
	Los Angeles, Calif. (Southern California) Roger Young Auditorium October 29, 1946	A Steel Selection Chart for the Materials Engineer by R. A. Schaus, and Application of Electric Strain Gages to Static and Dynamic Testing, by G. A. Brewer. Also, presentation of Templin Award. (83 in attendance)
	Cleveland, Ohio Cleveland Engineering Society Auditorium November 7, 1946	Testing Materials with the Atomic Bomb, H. N. Stevens; Progress in Rubber,[a] by President A. W. Carpenter. (100 in attendance)
	Detroit, Mich. Rackham Memorial Building November 13, 1946	Materials—Where are They, by A. R. Maupin[b]. (200 in attendance)
	Philadelphia, Pa. Franklin Institute November 14, 1946	Water for Industry, by L. D. Betz and J. J. Maguire. (200 in attendance)
	St. Louis, Mo. Park Plaza Hotel November 15, 1946	Jointly with A.S.M.E. Wood as an Engineering Material, by L. J. Markwardt. (65 in attendance)
	Boston, Mass. (New England) Huntingdon Hall (M.I.T.) (Cambridge, Mass.) November 21, 1946	First New England Technical Meeting. Experimental Stress Analysis, by W. M. Murray, A. C. Ruge, and G. Ellis. (300 in attendance)
	Pittsburgh, Pa. Mellon Institute Auditorium December 2, 1946	Testing Materials with the Atomic Bomb, by H. N. Stevens; Progress in Rubber,[a] by President A. W. Carpenter. (175 in attendance)
	New York, N. Y. Engineering Societies Bldg. December 12, 1946	The Future of Atomic Energy, by J. R. Dunning. (500 in attendance)
	Chicago, Ill. Engineering Bldg. Auditorium January 7, 1947	Testing Materials with the Atomic Bomb, by H. N. Stevens; Progress in Rubber,[a] by President A. W. Carpenter. (250 in attendance)
	Philadelphia, Pa. Franklin Institute January 23, 1947	Gas Turbines, by Russell Franks and F. W. Godsey, Jr. (capacity attendance of 450, with overflow)
	New York, N. Y. Engineering Societies Bldg. March 4, 1947	Quality Control, by G. C. MacDonald. (250 in attendance)
	Northern California (Berkeley) Claremont Hotel March 10, 1947	Jointly with A.C.S. Progress in Chemical Industry Through Research and Standardization,[c] by President A. W. Carpenter. (250 in attendance)
	Buffalo, N. Y. Western New York-Ontario Hotel Sheraton (Rochester) March 13, 1947	Gasoline—Yesterday, Today, Tomorrow,[d] by Vice-President T. A. Boyd; Rubber in Automotive Engineering,[e] by B. P. Resseger. (75 in attendance)
	Southern California (Los Angeles) Roger Young Auditorium March 18, 1947	Jointly with Los Angeles Rubber Group, Inc. Progress in Rubber, by President A. W. Carpenter; A.S.T.M. and Its Work, by Executive Secretary C. L. Warwick. (150 in attendance)

A.S.T.M. MEETINGS JUNE, 1946–MAY, 1947, INCLUSIVE—*Concluded*

Meeting	Place and Date	Notes
District or Local (sponsored by Councils Indicated) (Cont.)	Pittsburgh, Pa. Mellon Institute Auditorium May 19, 1947	Gasoline—Yesterday, Today, Tomorrow,[d] by Vice-President T. A. Boyd. (160 in attendance)
	New York, N. Y. Engineering Societies Bldg. May 27, 1947	Radar—The What, How and When of One of the Most Effective Weapons of World War II, by Captain Ernest G. Atkins. (160 in attendance)

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[a] Published in ASTM BULLETIN, No. 144, January, 1947.
[b] Abstract published in ASTM BULLETIN, No. 143, December, 1946.
[c] Published in ASTM BULLETIN, No. 146, May, 1947.
[d] Abstract published in ASTM BULLETIN, No. 146, May, 1947.
[e] Abstract published in ASTM BULLETIN, No. 146, May, 1947.

NOTE.—Paper by E. S. Lee on "What Is New In Science and Engineering," presented January 29, 1946 at Northern California District Meeting, published in ASTM BULLETIN, No. 141, August, 1946.

Year Book and that of Committee A-7 will appear in the 1947 edition.

The title of Committee B-4 was changed to read "Electrical Heating, Resistance, and Related Alloys" without change in scope.

The officers of Committees D-2 on Petroleum Products and Lubricants and D-3 on Gaseous Fuels have arranged that sections of Technical Committee H, of Committee D-2, on Light Hydrocarbons dealing with matters of common interest to the two technical committees shall function under the sponsorship of both and that the personnel of these sections shall have the approval of both committees.

DISTRICT ACTIVITIES

The administration of district activities is delegated by the Board of Directors to the Administrative Committee on District Activities, whose report of current developments is appended hereto (Appendix VII). A most important achievement in district affairs, as noted in that report, was the adoption by the Board of Directors of an extensively revised and amplified Charter for districts, as well as a Manual, the latter being essentially a compendium of good practice and a supplement to the Charter. The new Charter gives to districts a greater measure of autonomy and covers specifically a number of administrative matters which were only referred to or not mentioned in the earlier charter. One of the major changes provides that districts shall nominate and elect their own officers who are members of the local Council. This is in contrast with the earlier method of having district officers appointed by the Board of Directors.

The eleven districts through their Councils have cooperated in numerous ways during the past year and, as indicated below, have sponsored a number of technical meetings.

Special mention should be made of the way in which the Philadelphia District served as host during 1946 Committee Week and the Spring Meeting and aided in planning and in carrying out the functions incident to the official dedication of the Headquarters Building. The District Council arranged for the reception and the dedication dinner and underwrote all expenses in connection with the exercises.

The Northern California District arranged and manned an interesting booth display in the Western Metal Congress held in Oakland in March. This District is planning to participate in a similar manner in the Pacific Chemicals Exposition scheduled for October.

As noted in the May BULLETIN, the President and Executive Secretary were able to visit the Pacific Coast in March to meet with A.S.T.M. members in the Northern and Southern California Districts and to speak at meetings in San Francisco and Los Angeles. Numerous conferences were held during their stay. There is an enthusiastic group of members on the Coast which augurs well for a continuation of the interest in A.S.T.M. in this rapidly developing industrial area.

On the recommendation of the Administrative Committee, the Board of Directors has authorized the formation of a district in the area centered in Washington, D. C., probably to encompass the State of Maryland and parts of Virginia. Temporary appointment of Council personnel will be made prior to formal organization of the district.

District Meetings:

From the accompanying table of A.S.T.M. meetings, it will be noted that each of the districts sponsored one or more technical meetings. News accounts of these have appeared in the BULLETIN and abstracts or synopses of the papers or addresses have been included. President Carpenter spoke at several meetings, and two of his addresses were published in the BULLETIN.

Meetings which might be noted particularly were those in New York featuring the subject of Atomic Energy, and the Philadelphia meeting on Gas Turbines. The New England District held its first technical meeting, a very successful and interesting one. The districts have continued to extend meeting invitations to the members of other local sections and groups, frequently through the cooperation of these other bodies by mailing notices of the meeting direct to their members. It has been evident that a considerable percentage of the audiences have been non-A.S.T.M. members and in this respect the meetings have achieved one of their objects, namely, that of bringing a wider knowledge of the Society and its activities to the various localities in which the districts function.

The Board of Directors desires to record its appreciation for the loyal efforts and cooperation of the district committee officers and members. Many of them are active in other A.S.T.M. work, and the interest which they take in district work is indicative of the importance with which this is viewed by our members.

NATIONAL MEETINGS

The 1946 Annual Meeting in Buffalo during the week of June 24 was an outstanding one, with a larger number of symposiums held than at any previous meeting. The technical papers and discussions comprising these symposiums afford a great wealth of data and information, and the publications covering these symposiums, most of which have now been issued, have still further enhanced the Society's reputation as a source of significant information on the properties and tests of materials. The Seventh Exhibit of Testing Apparatus and Related Equipment held during the meeting and the Fifth Photographic Exhibit were important features of the meeting, both being very interesting and successful affairs.

The Spring Meeting in Philadelphia in February, 1947, featured a Symposium on Paints and Paint Materials, with over 500 in attendance at each of the two technical sessions. This symposium was arranged by Committee D-1 on Paint, Varnish, Lacquer, and Related Products, with the cooperation of the Philadelphia District. It is to be issued shortly as a special publication.

1948 Meetings:

The Board of Directors has decided to hold the 1948 Annual Meeting and the

Eighth Exhibit of Testing Apparatus and Related Equipment in Detroit during the week of June 21. Headquarters will be at the Book-Cadillac Hotel with other hotels, notably the Statler and Detroit-Leland, cooperating in furnishing sleeping rooms, committee rooms and other facilities.

Committee Week and the Spring Meeting in 1948 will be in Washington, D. C. during the week of March 1. The Statler Hotel will be headquarters for the meeting, with the Mayflower and other nearby hotels collaborating.

1949 Meeting on Pacific Coast:

The Northern and Southern California District Councils have joined in extending an invitation to the Board of Directors for the Society to hold a national meeting in San Francisco in October, 1949. On their recent visit to the Coast the President and Executive Secretary discussed this invitation with the two Councils and the Board is giving earnest consideration to the acceptance of the invitation. Tentative commitments have been made for dates and facilities in San Francisco and the Board's Committee on Meetings and Program is now considering several questions that are involved in the holding of a successful meeting on the West Coast.

PUBLICATIONS

Responsibility for the publications of the Society is delegated to the Administrative Committee on Papers and Publications, and the report of that committee attached as Appendix IV reviews the year's activities in this field and gives interesting information about the publications of the past year as well as plans for some future publications. The biggest single publication of the year is the new edition of the Book of Standards comprising about 6700 pages published for the first time in five parts. It is planned that this edition will cover the triennial period 1946–47–48, with Supplements thereto in the two latter years. The exceptionally large volume of technical papers submitted to the Society during 1946 has resulted in one of the largest volumes of annual Proceedings, together with a number of special publications. The ASTM BULLETIN has to an increasing extent served as the medium for publication of papers offered throughout the year.

The volume of principal publications for 1946 is by far the largest in the Society's history, exceeding 10,000 pages—nearly 2000 pages greater than the nearest comparable year, 1944. Details are given in Appendix IV.

The Special Technical Publication Series has now been established and a list of the publications in that Series is printed in the May BULLETIN. The Series comprises all important special publications issued from the beginning of the Society's history, and hereafter such publications will be made a part of the Series and numbered accordingly.

The Administrative Committee on Papers and Publications, as noted in its report, is engaged in two important studies: The first has to do with the future publication of the standards of the Society, with particular reference to whether it is practicable to continue the Book of A.S.T.M. Standards in its present form. The other study involves the methods of publishing the reports and technical papers of the Society, with particular reference to such matters as annual *versus* quarterly or monthly *Proceedings* and the relationship thereto of the BULLETIN. Some of the considerations involved in these studies are covered more fully in the Administrative Committee's report.

One of the most serious problems in publications today is the greatly increased cost and the difficulty of getting the ever increasing volume of our publications expeditiously printed. A number of the more important publications have been greatly delayed in reaching the members, which is vexatious and at times a serious inconvenience. Until recently three printing houses have handled the printing of the Society's principal publications and now an effort is being made to locate additional printing establishments equipped to handle some of our work.

Sam Tour Award:

The Board of Directors is pleased to announce that Sam Tour, head of the firm of Sam Tour and Co., Inc., and a Director of the Society, has made a gift to the Society of $1500 to be used for the establishment of an award for the purpose of stimulating papers on corrosion testing. In making the gift Mr. Tour refers to his twenty-five years of activity in the Society in the field of corrosion, which coincides with the life of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, of which he was the first secretary for a period of eighteen years and chairman for the next four years. With his gift he pointed out how little we still know of corrosion testing and how important it is that more be known on the subject, and he has expressed the hope that the establishment of this award will direct more specific attention to the development and evaluation of corrosion testing methods.

The Board of Directors has been pleased to accept this gift and with it has established the Tour Award to be, as the donor suggested, in the form of a certificate of merit, a medal, or a monetary award as the Award Committee may determine from time to time. The Board has appointed a special committee to prepare rules governing the award which will be announced to the membership through the ASTM BULLETIN.

ADMINISTRATIVE MATTERS

Revision of By-laws:

The Board of Directors recommends to the Society two amendments in the By-laws, as follows:

Article III, Section 2, Nomination of Officers.—This section deals with the procedure for obtaining recommendations for members of the Nominating Committees and the dates fixed therein were determined by the date at which the former Executive Committee held its fall meeting, namely, October. The new Board of Directors has revised the schedule of meetings so that its fall meeting will be held about the middle of September. The advance of a month in this meeting date makes it desirable to advance correspondingly the two dates in this section of the By-laws: The first is the date of September 1 by which the Executive Secretary shall have notified members of the Society of the right to recommend two members for appointment on the Nominating Committee, and it is recommended that this date be changed to July 1. The second date, October 1, is the date by which returns must be received by the Executive Secretary in order to be canvassed by the Committee of Tellers, and it is recommended that this date be changed to August 15.

Article VIII, Section 1, Dues.—As discussed elsewhere in this report, the Board of Directors is recommending for the consideration of the Society that the annual dues be increased and that accordingly Section 1 of Article VIII be changed from its present form to read as follows:

> *Section 1.*—The fiscal year shall commence on the first day of January. The annual dues, payable in advance, shall be as follows: For Individual Members, $18; for Company Members, $50; for Sustaining Members, $150; for Junior Members, $9; for

Student Members, $2. Honorary Members shall not be subject to dues.

Administrative Organization:

During the year the final steps were taken to complete the reorganization of administrative activities that have been discussed in the two previous reports. The new Board of Directors took office at the close of the last annual meeting and held its organization meeting at Buffalo on June 28, 1946. At this meeting appropriate actions were taken to meet legal requirements involved in the transfer of authority from the former Executive Committee to the new Board, particularly with reference to financial matters and the administration of the A.S.T.M. Retirement Plan.

The Board also adopted a set of Regulations Governing the Board of Directors, including rules governing the conduct of Society finances, which are published in the 1946 Year Book, pages 472–475.

The Board continued the six Administrative Committees that had previously been established, namely, on Standards, Research, Papers and Publications, Ultimate Consumer Goods, Simulated Service Testing, and District Activities. It also appointed seven standing committees of the Board, namely, Executive Committee, Finance, Membership, Meetings and Program, Technical Committee Activities, Inter-Society Relations, and Developmental Activities. The Executive Committee is a new committee for which provision was made when the By-laws were amended in 1945 to give effect to the plans for administrative reorganization. The Board has constituted the Executive Committee to comprise the senior Vice-President as chairman, the junior Past-President, the President, and three Directors and has delegated to it certain authorities in the administration of the Society affairs. While this year has been one of transition between the old and the new administrative procedures, it is already clear that the Executive Committee can take care of much routine business, leaving the Board freer to devote its attention to broad questions of policy in the administration and functioning of the Society.

No further changes in the organization of the headquarters staff were made during the year, although the growth of activity necessitated an increase of eight persons in the stenographic and clerical staff. There are now eleven persons in the administrative staff and 32 in the stenographic and clerical, or a total of 43, compared with 35 a year ago. In addition to the regular staff, there are four persons presently employed on a temporary part time basis.

Society Headquarters:

The new Society Headquarters have been occupied for about a year, although it was not until the late fall of 1946 that the alterations were completed and the building substantially finished except for installation of air-conditioning units. There has been serious delay in this portion of the work, but there are now indications that this will be completed during the summer of 1947.

The building was formally dedicated on Wednesday, February 26, 1947. There were appropriate exercises at the building in the afternoon, followed by a Dedication Dinner at the Benjamin Franklin Hotel in conjunction with the Spring Meeting. A full account of these exercises, together with a description and photographs of the building and various offices appeared in the March, 1947, ASTM BULLETIN.

The following inscription, which was unveiled at the dedication exercises, appears on one of the walls of the vestibule:

"This building was given by the members to the Society as national headquarters and is dedicated to the advancement of education and science and to service to industry and to the nation."

The building has proved to be well adapted to the Society's needs and the Board of Directors has been pleased to learn of the many favorable comments on the new headquarters that have been offered by our members and visitors. It is particularly gratifying to record that the meeting room facilities are being used by our committees and it is hoped that committees and the membership generally will make the fullest possible use of the headquarters building.

Financial Aspects.—The total cost of the headquarters, including purchase of land and building and all alterations, will be approximately $184,000. The total of contributions to the Building Fund up to June 1, 1947 , is $156,045; other additions to the Fund, principally interest and profit from sale of investments, bring the total income to approximately $157,000, so that to own the building clear of indebtedness, which in the opinion of the Board of Directors would be highly advantageous to the Society, will require further additions to the Fund of approximately $27,000. Contributions to the Fund may be made at any time, and the Board suggests that there are many members who have not so far contributed who may now wish to do so.

The Board has acted favorably upon a suggestion, made by one of the technical committee chairmen, that the committees of the Society be invited to contribute to the Building Fund through subscriptions of the committee members obtained during the course of the coming year. It is believed that many of the committees will welcome this opportunity of aiding the Society in reaching the desired goal.

MEMBERSHIP

On June 1, 1947, the membership of the Society totaled 6313, the highest figure yet reached, compared with a total of 5993 on June 1 one year ago. Statistics showing gains and losses in various classes of membership are shown in the following table:

Class of Membership	Membership		Losses				Additions		Total		
	June 1, 1946	June 1, 1947	Resignations	Dropped	Death	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	9	14	...	..	1	..	5	1	1	6	5
Perpetuity and Life	10	12	...	..	..	..	2	..	..	2	2
Sustaining	194	213	1	..	..	..	19	1	1	20	19
Company, Firm, etc.	1581	1650	59	4	..	21	6	147	84	153	69
Individual, etc.	4089	4305	182	49	36	13	4	492	280	496	216
Junior	110	119	10	11	..	2	..	32	23	32	9
Total	5993	6313	252	64	37	36	36	673	389	709	320
Student	138	336	7	63	..	12	..	280	82	280	198

The net gain for the year of 320 compares with 344 for the preceding year. Elections to membership were the highest in the history of the Society and numbered 673, exceeding those for the previous year by 19; losses from resignations and delinquencies were 316 compared to 281 for the preceding year. Junior Members numbered 119 and Student Members 336.

There have been twenty new sustaining members as indicated below, which with one resignation brings the total to 213:

American Bemberg Corp.
The Bristol Brass Corp.
Canadian and General Finance Co., Ltd.
Concordia-Gallia Corp.
Douglas Aircraft Co., Inc.
The General Tire and Rubber Co.
Hartford-Empire Co.
Hughes Tool Co.
S. C. Johnson and Son, Inc.
R. G. LeTourneau, Inc.
Lima Locomotive Works, Inc.
Arthur D. Little, Inc.
Mohawk Carpet Mills, Inc.
The Okonite Co.
Richfield Oil Corp.
John A. Roebling's Sons Co.
Stone & Webster Engineering Co.
Taylor Instrument Cos.
Tube Turns, Inc.
R. T. Vanderbilt Co., Inc.

Honorary Memberships:

The Board of Directors has elected to Honorary Membership in the Society the following three members: F. Malcolm Farmer, Vice-President and Consulting Engineer, Electrical Testing Laboratories, Inc., New York City; Arno C. Fieldner, Chief, Fuels and Explosives Service, U. S. Bureau of Mines, Washington, D. C.; and Hervey S. Vassar, Laboratory Engineer, Public Service Electric and Gas Co., Maplewood, N. J. They will be presented to the Society for formal award of honorary membership during this Annual Meeting.

40-Year Members:

In 1938 the Executive Committee established the plan of awarding at annual meetings certificates commemorating forty years of continuous membership in the Society. One hundred such certificates have thus far been awarded and this year the following 14 companies and individuals have qualified:

Herbert Abraham
E. H. Beckstrand
Henry C. Boynton
Alexander J. Christie
Thomas R. Lawson
C. P. Marsh
Raymond G. Osborne
Reading Company
Rensselaer Polytechnic Institute
Harry A. Schwartz
John Fairfield Thompson
Percy H. Walker
Morton Owen Withey
Youngstown Sheet and Tube Co.

Deaths:

The Society has lost the following 37 members through death:

	Date of Membership
Agg, Thomas R.	1914
Allinson, J. J.	1932
Ball, George L.	1941
Boniface, A. O.	1944
Bradbury, Royall D.	1930
Burwell, Arthur Warner	1933
Cross, Roy	1916
Cutts, Henry E.	1936
Dengler, F. S.	1922
Forrest, Charles N.	1901
Fulks, E. B.	1925
George, W. J.	1943
Grosch, H.	1946
Haring, W. S.	1938
Herr, J. C.	1935
Hutchinson, George W.	1937
Jordan, Guy W.	1939
Kellermann, William F.	1938
Knopel, Herbert J.	1935
Krenzke, William F.	1943
Lamborn, Samuel W.	1944
Malleis, O. O.	1916
Miller, Rudolph Philip	1903
Oller, John	1921
Pew, J. Edgar	1923
Ramsay, John	1924
Rice, Kenneth	1941
Seil, Gilbert E.	1935
Shepard, Robert B.	1935
Shirk, Frederic Krauter	1939
Shotton, Bruce G.	1942
Smith, Harold De Witt	1938
Tatlock, James Douglass	1924
Thieme, Carl O.	1920
Toch, Maximilian	1903
Todd, G. H.	1937
Trautman, H. A.	1944

In addition to the above, the following representatives of company members of the Society passed away:

C. Huddleston Bear, Oxychloride Cement Assn., Inc.
W. G. Bjorkstedt, International Projector Corp.
A. G. Fleming, Canada Cement Co.
O. C. Hart, Monolith Portland Cement Co.
Robert Moore, Jr., Bakelite Corp.
H. L. Randall, Riverside Metal Co.
James A. Smail, Republic Steel Corp.

A number of these men, several of whom have been connected with the Society continuously for thirty or more years, have rendered notable service in connection with the Society's activities particularly in the work of technical committees. Charles N. Forrest, a former member of the Executive Committee, served for over forty years and was a leader in the work of committees in the field of road and paving materials and bituminous waterproofing and roofing materials. Guy W. Jordan had many years of service on Committee C-1 on Cement. O. O. Malleis had a notable record of service with Committee D-5 on Coal and Coke and its related activities. Rudolph P. Miller, one of the country's outstanding authorities in the field of building code work and fire tests of building construction and materials, was a long-time chairman of Committee C-5 (now E-5), and his services in A.S.T.M. and his eminence were recognized by his election to honorary membership. Dr. Harold De Witt Smith, an eminent scientist in the field of textiles and a member of Committee D-13, delivered the A.S.T.M. 1944 Edgar Marburg Lecture. Carl O. Thieme had been concerned with our work in the field of chemical analysis of metals, and Maximilian Toch was for many years active on Committee D-1 on Paint, Varnish, Lacquer and Related Products. A. G. Fleming was for many years concerned with the work of Committee C-1 on Cement and served in other groups. The death of Robert Moore, Jr. came when he was serving A.S.T.M. as Chairman of the Administrative Committee on Research; he had an inspiring record of work on Committees D-1 on Paint, Varnish, Lacquer, and Related Products, D-9 on Electrical Insulating Materials and D-20 on Plastics.

Some Membership Comments:

Under the guidance of the Committee on Membership headed by W. C. Hanna, which group has taken a very direct and inspiring interest in membership promotion, a number of activities have been carried out during the year, all of which have been reflected in the membership growth. This committee contacted quite a number of company members suggesting the possibility of a transfer to sustaining membership, and the number of new sustaining members listed above resulted. Contacts have been made with the officers of A.S.T.M. technical committees and will be made with the District Councils through the Administrative Committee on District Activities, suggesting that each of these groups consider membership promotion. Committee D-1 on Paint, Varnish, Lacquer, and Related Products set up a subcommittee on membership some time ago and the results have been very worthwhile.

The committee is interested in stimulating more student memberships and plans to enlist the interest of deans of engineering schools in this particular class. For the small yearly dues a student member gets a great amount of material, much of which can be of help to him in his studies, and he can become familiar with important developments which are covered in technical papers and reports.

Finances

Report for the Fiscal Year 1946:

The annual statement of the finances of the Society is presented in the form of the report of the auditors for the fiscal year January 1 to December 31, 1946, which appears in Appendix I. The report gives the balance sheet of assets and liabilities, including general funds, building fund, and other special and designated funds; a statement of receipts and disbursements classified into "operating" (budgeted) and "nonoperating"; details of the building fund and of special funds; and investments of Society funds.

Total operating receipts were $312,083.42, which were exceeded only by those of the preceding year. Of this amount receipts from dues and entrance fees were $136,877.16 or 43.9 per cent; receipts from sales of publications were $142,231.05 or 45.5 per cent; receipts from miscellaneous sources, consisting principally of advertising, interest and dividends, registration fees, and exhibit totaled $32,975.21 or 10.6 per cent.

Operating disbursements with all current bills paid, but including only about half of the estimated cost of the current Book of Standards, were by far the highest in the Society's history, totaling $339,994.53. This is approximately $63,000 higher than the next highest year, 1945, which is accounted for principally by two things: first, the greatly increased costs of operations, especially printing and related services essential to the Society's work, and including "cost of living" increases in salaries made during the year; and second, expansion of technical activities, with concomitant increases in size and number of publications and in staff personnel. Approximately 20 per cent of the $63,000 is represented by increased publication costs, 35 per cent by increase in the salary roll including additions to the staff, and the remainder by increases in office expenses, committee and meetings expenses, travel expense, and furnishings.

The difference between operating receipts and disbursements constitutes an operating deficit of $27,911.11, which is about 9 per cent of receipts. To meet this deficit there were applied to current operations the entire Headquarters Reserve of $6491.56 to cover moving expenses and a part of the cost of new furnishings; the sum of $1250.85 from the Publication Reserve to meet the cost of the work of the Joint Committee on X-Ray Diffraction; and $600 from the Retirement Fund Reserve to go towards the Society's contributions to the Retirement Plan,—leaving a balance of $19,568.70, which was taken from surplus.

The auditors' report includes a statement of the financial position of the Society for the past five years. The surplus in the General Funds account on December 31, 1946, was $144,210.75, compared to $165,037.47 a year ago. The ratio of surplus to annual operating disbursements at the current level is approximately 35 per cent, which is the lowest for some years. The Board of Directors feels that this is too low a ratio for sound operations, and that the surplus should be at least 50 to 60 per cent of annual operating expenses to provide adequate reserves for initiation of new activities and to meet emergency conditions.

The auditors' report also includes a statement of cash receipts and disbursements in the Building Fund as well as a balance sheet for the Fund as of December 31, 1946. As previously noted in this report, it is expected that alterations to the Headquarters Building will be completed this year and final costs established, after which the building will be carried on the balance sheet as an asset at full cost with provision for annual depreciation of the building and amortization of the Fund beginning with 1947.

Investments of Society funds as of December 31, 1946, are given in the auditors' report, both book and market values being shown. The following changes during the fiscal year in the investment portfolio are noted:

GENERAL FUNDS

Bought	Cost
$5 000 Walworth Co. Conv Deb. 3¼'s, 1976	$5 496 53
22 shares American Airline Inc, 3½ Cum. Conv. Pfd.	2 244.00
30 shares American Viscose Corp., Common	2 323 58
100 shares Baldwin Locomotive Works, Common	3 822 00
100 shares Carpenter Steel Co., Common	5 426 00
100 shares International Harvester Co., Common	9 610 00
60 shares Lone Star Cement Corp., Common	4 349 42
20 shares Philco Corp. Series A 3¾ Pfd.	2 030 00
	$35 301 53

Sold, Called or Matured	Received
$2 000 Columbia Gas & Electric Co. 5's, 1961	$2 040 00
22 000 U. S. Treasury Note B, 1947	22 097 45
25 shares Hazel Atlas Glass Co.	3 995 38
20 shares G. C. Murphy Co. 4¾ Pfd.	2 190 82
	$30 323.65

RESEARCH FUND

Bought	Cost
30 shares Allis Chalmers Mfg. Co. 3¼ Cum. Conv. Pfd.	$3 007.22
33 shares American Airlines Inc. 3½ Cum. Conv. Pfd.	3 366 00
100 shares Ohio Oil Co., Common	2 167 88
20 shares Philco Corp. Series A 3¾ Pfd.	2 030 00
	$10 571 10

Sold, Called or Matured	Received
$2 000 Columbia Gas & Electric Co. 5s, 1961	$2 040 00
3 000 U. S. Treasury Note B, 1947	3 002 81
30 shares G. C. Murphy Co. 4¾ Pfd.	3 286 24
	$8 329.05

In the General Funds, profits on securities sold, called or matured totaled $1638.35; in the Research Fund profits from the same sources totaled $153.74.

No account has been taken in the accompanying financial statement of the assets of the Society in the form of publications in stock. An inventory of technical publications as of May 1, 1947, may be summarized as follows:

	Number of Copies
Year Book (1946)	1 450
Proceedings	2 978
Index to Proceedings (total)	974
1946 Book of A.S.T.M. Standards:	
Part I A	5 151
Part I B	5 405
Part II	4 124
Part III A	3 456
Part III B	3 434
A.S.T.M. Methods of Chemical Analysis of Metals	2 739
Index to A.S.T.M. Standards (1945)	3 321
X-Ray Diffraction Cards, sets	88
Compilations of Standards	8 080
Selected Standards for Students	1 000
Symposiums and Special Reprints	48 436
Reprints of Standards (approximate)	100 000

1947 Finances:

The problem of budgeting the Society's operations for 1947 is a difficult one. Provision for expanded technical activities is essential, involving new and larger publications and certain increases in staff. The Board has been prepared to draw for this purpose upon surplus, believing that in due time these activities will become self-supporting through increased memberships and increased publication sales. However, the greatly increased costs of carrying on the Society's work, previously mentioned, coming just at the time of expanded activities, have created a serious financial problem. This problem was recognized a year ago, and since increased cost of printing is one of the largest factors involved, immediate steps were taken to augment income by increasing the selling prices of Society publications. The publications principally involved were the 1946 edition of the Book of Standards, the various Compilations of Standards, and all special publications issued after the last annual meeting. The new prices reflect both the increased printing and handling costs, and the greater intrinsic value of all publications containing standards, of which there are many new and revised. At the same time, the charges to members for procuring more than one part of the Book of Standards and its Supplements were increased, effective January 1, 1947. Rates for advertising in the BULLETIN and Index to Standards were also increased, bringing them generally into line with rates for similar publications.

These various changes had little effect on 1946 income, and with mounting costs during the latter part of that year an operating deficit for 1946 resulted, as already reported.

Giving full effect to increased income from these sources for 1947, estimating publication sales as optimistically as seemed wise, and assuming further that there would be no serious reversal of trend in growth of Society income such as could result from an economic depression, operating receipts for 1947 were estimated at $401,000. Admittedly, a serious business recession could cut into this estimate, and such is entirely possible. However, certain items have been quite conservatively estimated, so the current budget was set up with $401,000 for receipts.

Society budgeting needs to be considered over a three-year period, coinciding with the triennial publication of the Book of Standards, and since the sale of the Book of Standards is much greater in the first year and drops off markedly in the second and third years, normally a favorable operating balance will result in the first year that can be applied to probable operating deficits in the second and third years of the budget period. This was the experience, for example, in the last normal prewar period, 1937–38–39; then the war with its vastly accelerated production and demand for all types of technical information, especially standards, introduced factors that changed expected deficits in 1941 and 42 to surpluses, and led to two biennial publication periods (1943–44 and 1945–46) with favorable operating balances in all but the last year, 1946.

Now, at the start of the triennial period 1947–48–49, a favorable operating balance for 1947 should be obtained; but when disbursements were estimated for the year's operations, with provision for publications, salaries, committee work, meetings, and general operations as required and for the very minimum of staff expansion, it was seen that about $406,000 would be required, resulting in a small deficit instead of the hoped-for favorable balance. Expenses have been very closely figured, and while no technical publications have been curtailed despite the high printing costs, a substantial economy of about $9000 was effected in eliminating expensive cloth binding of the five 1947 Supplements to Standards, substituting heavy paper covers. The Board would have liked to provide more liberally for expansion of staff more effectively to take care of growing technical and publication activities, but concluded it was not wise to do so in the face of a deficit and with reduced surplus as previously noted. Accordingly, disbursements were fixed at $406,000, and the budget was balanced by applying $5000 from reserve funds and surplus to cover estimated expenses of the X-Ray Diffraction Committee ($2000), certain expenses of the Retirement Fund ($500) and cost of prepaying fire insurance to obtain a 20 per cent saving in premium ($2500).

The budget as thus established last January was reviewed by the Board after the first quarter's operations and no changes were considered necessary. Experience thus far in 1947 confirms the general assumptions upon which the budget has been based. Periodic reviews will be made throughout the year.

To complete reference to 1947 finances thus far in the year, it is noted that no securities have been sold this year and the following securities have been bought: 16 shares of U. S. Gypsum Common (through stock warrants) for the Research Fund; and 50 shares of Montgomery Ward and Co. Common, of which 30 shares are for the General Fund and 20 shares for the Research Fund.

The total book value of Society investments as of May 31, 1947, in all funds was $237,308.38. The Society's cash balance on May 31,1947, was $140,822.10.

1948 and Beyond:

Looking ahead, no favorable balance in 1947 for carry-over into the next two years of the budget period can safely be counted upon. The best estimate that the Board of Directors can make at this time is that, with income in sight at present dues and maintaining current levels of activity, we face an operating deficit in 1948 of the order of $30,000 to $40,000 and it might well be greater under adverse business and economic conditions. The year 1949 presents a similar picture so far as it can be seen from this distance. This estimate is based on experience of the past two post-war years and assumes continuance of present cost levels; it gives full effect to sources of increased income established in publication sales and advertising as previously noted; it provides for the growing volume of reports and standards flowing from our expanded committee work, but not for adequate increase in staff to implement the Society's program of post-war work and development. To keep expenditures within the estimated income could only be done by curtailment of Society activities and publications, which means less service to the members and to industry, and to all other groups that today make such effective use of the results of the Society's work. The Board of Directors believes that such curtailment would be a backward step and that the membership would not wish to adopt such a policy.

Obviously the Society cannot incur deficits and continue to operate on a sound financial basis. Expressed in another way, the costs of the services that the Society has been giving to the membership in ever-growing measure have so increased that they are no longer adequately supported by present income. Since income from sources other than membership dues already constitutes about 60 per cent of total income and it is believed cannot be further raised to such levels as to meet the pending operating deficits, the Board of Directors has concluded, after most careful consideration of the entire matter, that we must look to an increase in membership dues to provide the added funds required for operation of the Society.

Increase in Dues Recommended:

Accordingly the Board recommends to the membership that, effective January 1, 1948, the membership dues be increased in the several classes of membership as follows:

Class of Membership	Present Dues	Proposed New Dues
Sustaining	$100	$150
Company	30	50
Individual	15	18
Junior	7.50	9
Student	1.50	2

This will require that Article VIII, Sec. 1, of the By-laws be amended to read as follows:

> *Section 1.* The fiscal year shall commence on the first day of January. The annual dues, payable in advance, shall be as follows: For Individual Members, $18; for Company Members, $50; for Sustaining Members, $150; for Junior Members, $9; for Student Members, $2. Honorary Members shall not be subject to dues.

This proposed amendment was printed in the notices for the Annual Meeting (Letter Circular to Members No. 274, May 26, 1947), as required by the By-laws and will be presented at a business session during the annual meeting at which, in accordance with the provisions of the By-laws, it "may be discussed and amended and may be passed to letter ballot by a two-thirds vote of those present." Such a letter ballot of the Society membership, authorized at this business session, is necessary to amend the By-laws and requires a two-thirds affirmative vote for adoption.

In considering the question of dues a complete realignment of the present membership and dues structure could have been set up, but the Board concluded that the present classes of membership are well suited to the needs and activities of the Society and should be retained. The proposed new scale of dues is designed to bring about greater support of Society activities by industry as represented by sustaining and company members, at dues that are more nearly commensurate with the value of the service that the Society renders to industry. It is realized that industry supports the work of the Society very extensively through its participation in committee work and that the annual dues of a company frequently are a very minor part of its total contribution to A.S.T.M. However, greater direct support from industry into the treasury of the Society is essential if we are to maintain an organization that will enable us to do all that we are being called upon to do for the members and for industry generally.

The dues for individual members, comprising persons, colleges and universities, technical societies, libraries, government agencies, etc., are increased only a nominal amount, which the Board believes is a desirable policy to follow with respect to this class of membership, as well as for junior and student members.

The entire proposal is premised on the present publication policies of the Society. As indicated elsewhere in this report these policies are under careful study by the Committee on Papers and Publications. It is entirely possible that in the future some important changes may need to be made in methods of publishing standards, reports, and papers. However, it is not expected that any of the changes that might be made would be of such a nature as to affect the dues structure, although some adjustments in charges for publications might be involved.

It is estimated that the proposed new dues will, at the current membership level and with allowance for adjustments incident to the change, result in an immediate annual increase in income of from $35,000 to $50,000 and in the future, in conjunction with other sources of income, will maintain the Society's financial position on a sound basis.

The Board in conclusion would express the thought that it is a prime responsibility of each of us as members, individually and collectively, to seek to advance the purposes for which the Society was organized just forty-five years ago—"The promotion of knowledge of the materials of engineering, and the standardization of specifications and the methods of testing"—and to provide the organization and the means for accomplishing these purposes to the very best of our ability. We are fortunate in being able so greatly to augment our income through various sources outside of membership dues. It is believed, however, that the analysis just presented leads logically to the conclusion that greater direct financial support from the membership itself is necessary to the continued attainment of our purposes, and the Board earnestly recommends that the proposed amendment be adopted by the Society.

Retirement Plan for Employees:

The Retirement Plan for Employees, begun in 1938, continues to develop in accordance with principles then established and subsequently extended and amended in 1944.[1] A consilidated statement of the Plan, embodying the provisions of all previous statements and of resolutions of the former Executive Committee and the present Board of Directors, was prepared as of October 1, 1946, and distributed to all members of the staff.

During the year, five newly eligible employees joined the Plan, bringing the number of participants to 24, out of 30 eligible employees. In addition, a number of new retirement policies were taken out to bring employee contributions up to 2½ per cent of salary and, through additional contributions by the Society, to bring retirement income values closer to the specified retirements for the respective employees, that is, one per cent for each year of service of average salary based on five highest years, up to $5000.

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[1] See *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 59 (1944).

The Society's total contribution in premiums for retirement policies for the above mentioned 24 employees is at the annual rate of approximately $10,000, which is roughly 7 per cent of the present salary roll and 2½ per cent of estimated current disbursements. The total of employees' annual contributions, including insurance benefits which most of them have elected, is approximately $3000.

COOPERATIVE ACTIVITIES

Cooperation with the Federal Specifications Board and its various technical committees has been continued along the lines indicated in last year's report. Contacts at the staff level are maintained and a recent ruling of the Federal Specifications Board has opened up the possibility of direct representation of Federal specification committees upon the committees of the Society. Such representation will not affect the extensive representation of the individual departments of the Government on our committees but will helpfully supplement Government participation in this committee work.

The Industry Advisory Council, which serves in an advisory capacity to the Federal Specifications Board and upon which the Executive Secretary serves as a representative of A.S.T.M., has during the year considered several matters of policy concerning which it has submitted recommendations to the Federal Specifications Board. The Council met jointly with the Board in Washington on May 16, and it accepted an assignment to advise the Board concerning the Proposed Manual for Federal Specifications. It has further been arranged that joint meetings of the Board and Council will be held twice a year.

This Council has also accepted the responsibility of serving in an advisory capacity to the United States Standards Commodity Catalog Board. One meeting has been held jointly with that Board at which a number of problems involved in the development of a comprehensive federal commodity catalog were discussed. Semi-annual meetings with this Board are also planned.

The Board of Directors has accepted an invitation from the Ordnance Department of the Army to appoint an A.S.T.M. Ordnance Advisory Committee. Conferences with the Chief of Research and Development Service of the Ordnance Department have indicated that there are ordnance problems in the field of materials related to the work of A.S.T.M. but not appropriately included in the agenda of our regular technical committees concerning which the proposed advisory committee can effectively bring to the service of the Ordnance Department the knowledge and experience of Society members and the various committee and other facilities of A.S.T.M. The services of the advisory committee can thus effectively supplement the extensive participation of the Ordnance Department in the work of many of our committees. The personnel of the committee is now being determined in consultation with the Ordnance Department; formal appointments will be made shortly and the committee organized in the fall in Washington, at which time the members will confer with members of the technical staff of the Research and Development Service of the Ordnance Department. Further announcements of the formation of the committee will be made in the ASTM BULLETIN.

The headquarters staff has maintained

interesting and worth-while contacts during the year with the Civil Aeronautics Administration of the Department of Commerce and the Naval Ordnance Laboratory. Each of these organizations has special interests in the work of the Society and it is mutually advantageous to maintain staff contacts. Arrangements are being made for representation of the Naval Ordnance Laboratory on certain of the Society's committees.

Conferences have been held at various times with officials of the Department of Commerce and its National Bureau of Standards concerning such matters as development of relationships between the Department of Commerce and Industry in the broad field of standardization, as proposed in the report of the Policy Committee on Standards discussed in the report a year ago. These discussions have included the relationship of the Society to the Government's work in the field of commercial standards and simplified practices.

H. F. Dodge of Bell Telephone Laboratories and A. E. R. Westman of Ontario Research Foundation, who are, respectively, chairman and vice-chairman of Committee E-11 on Quality Control of Materials, have been named as the Society's representatives on the Joint Committee on Statistical Applications in Engineering and Manufacturing, succeeding A. G. Ashcroft and W. H. Fulweiler.

J. T. MacKenzie of American Cast Iron Pipe Co., a Director of the Society, represented the Society at the 75th Anniversary Celebration of the American Institute of Mining and Metallurgical Engineers in March, 1947.

H. J. Gough of Lever Brothers and Unilever, Ltd., England, has been appointed by the Board to represent the Society at the Centenary Celebration of the Institution of Mechanical Engineers in London, June, 1947.

American Standards Association:

There have been no significant developments in the Society's relationships with the American Standards Association during the past year. The report of the Administrative Committee on Standards records a number of approvals of A.S.T.M. specifications and methods as American Standard. The Board of Directors, acting for the Society, cast an affirmative vote on a revision of the ASA Constitution, which provides for delegation of authority by the ASA Standards Council to a designated agency, such as the correlating committees of the Council, for formal adoption of American Standards.

The Company Member Committee of the American Standards Association, comprised of representatives of many of the company members of ASA, has been quite active during this year. One of the functions of the committee is to discuss industrial standardization activities to the end that the company members may be in a position to make recommendations to sectional committees, correlating committees, or the Standards Council of ASA regarding standards or standardization projects. During the year the Company Member Committee has discussed several questions that are of interest to the Society, including a proposal for unification and designation of properties of metals, the size to be used in the publication of standards, and the form and arrangement of standards. Representatives of the Society are participating appropriately in these discussions.

The Conference of Staff Executives of Member Bodies of ASA, of which the Executive Secretary is chairman, held one meeting during the year at which the subject of International Standardization was discussed from the viewpoint of the interest therein of the various member bodies. This meeting was held in conjunction with the annual meeting of ASA in November, 1946.

F. M. Farmer has been re-appointed as one of the Society representatives on the ASA Standards Council for a term of three years. J. R. Townsend continues as a second member of the Council, J. H. Foote has been named as a second alternate, with the Technical Secretary continuing as an alternate.

The following appointments have been made to committees of the ASA: J. R. Townsend to the Mechanical Standards Committee, A. G. Ashcroft to the Advisory Committee on Ultimate Consumer Goods, Miles N. Clair to the Building Code Correlating Committee, and Myron Park Davis to the Safety Code Correlating Committee.

International Activities:

During the year the International Organization for Standardization (ISO) has been formed, succeeding as an international body the former International Standards Association and taking over the work of the United Nations Standards Coordinating Committee which was established during the war. Some of the steps leading up to these developments were outlined in the report a year ago. A Constitution and Rules of Procedure for ISO were agreed upon at an international conference in London, October, 1946, and shortly thereafter the ASA. Board of Directors ratified the Constitution and Rules of Procedure on behalf of the American Standards Association, including the provisional organization of the ISO that had been effected in London, with the understanding that the other member bodies of ISO would be informed that the ASA plans to submit in due course certain changes in the Rules of Procedure, and possibly in the Constitution, in order to remove certain inconsistencies and bring about improvements. Mr. Howard Coonley, Chairman, ASA Executive Committee, has been elected the first president of ISO. The headquarters of the organization are being established in Geneva, and the first meeting of the ISO Council is to be held there in June, 1947.

The Board of Directors, acting for the Society, has expressed its support of the general objectives of ISO, with particular emphasis upon the function of coordination of various national standards throughout the world, rather than the initiation and writing of international standards *per se*. The Board, however, has taken exception to certain provisions in the Constitution and Rules of Procedure of ISO and has presented its views together with suggestions for changes in a communication to the ASA. It is understood that other member bodies have also offered suggestions and comments regarding the ISO organization.

In the meantime, the United Nations Standards Coordinating Committee is serving as the provisional secretariat in the handling of proposals for setting up new projects or the reestablishment of international projects that were under way in the ISA when the affairs of that organization were interrupted by the war.

The American Standards Association in this connection has received proposals from the ISO for initiating a number of standardization projects, and since some of these appear to lie within the field of interest and activities of A.S.T.M., the Board is endeavoring to obtain some precise information, including scopes of these projects, in order to determine the extent to which we might wish to participate in any of these projects. In anticipation of A.S.T.M. participation in such projects, the Board of Directors has established a procedure for determining the interest of the Society in any given project, which involves consultation with the appropriate A.S.T.M. committees. Consideration is also being given to the establishment of definite procedures governing the relationships between ASA and its member bodies in the handling of international standardization projects.

Contacts are being maintained with the Engineers Joint Council and with its Committee on International Relations concerning plans for the establishment of a World Engineering Conference. The Society is also following the work of the United Nations Educational, Scientific and Cultural Organization (UNESCO). It is thought that certain activities in UNESCO, particularly in the field of natural sciences, may be of interest to A.S.T.M. in connection with international discussions in the field of materials.

An International Conference on Soils Mechanics and Foundations is to be held in Holland in 1948. The Society's Committee D-18 on Soils for Engineering Purposes is expected to have an opportunity to help shape the program of American participation in this conference through the fact that a number of its members are serving on the American Conference Committee.

The Board reports that it has regretfully accepted the resignation of one of the Directors, Mr. A. T. Chameroy of Sears, Roebuck and Co., whose change in responsibilities within the company made it impossible for him to continue to serve. To fill the unexpired term to June, 1949, the Board has appointed Gerald C. MacDonald, Manager, Quality Control Group, Montgomery, Ward and Co., who has accepted the appointment.

Respectfully submitted on behalf of the Board of Directors,

ARTHUR W. CARPENTER,
President.

C. L. WARWICK,
Executive Secretary.
June, 1947.

APPENDIX I

REPORT OF THE AUDITORS FOR THE FISCAL YEAR, JANUARY 1 TO DECEMBER 31, 1946

Philadelphia, January 10, 1947

MR. C. L. WARWICK, *Executive Secretary*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pennsylvania

Dear Sir:

We have examined the books and accounts of the American Society for Testing Materials for the year ended December 31, 1946. We did not make a detailed audit of all transactions, but made extensive tests to determine the accuracy of the records. Accounts receivable were not verified by direct correspondence with debtors. The investments owned by the Society, were examined by us; all income from investments was properly accounted for.

No provision has been made for depreciation on the cost of building acquired in 1945 and the construction costs in relation to alterations and additions to such building expended largely in 1946. Such action is in accord with the instructions of the Executive Committee of the Society, as contained in the record of minutes of its meetings held June 21 and June 22, 1946, under which action depreciation is not to be taken into account until the period beginning January 1, 1947.

We have prepared and submit herewith balance sheet as of December 31, 1946, statement of cash receipts and disbursements for the year ended that date, and other supporting schedules, all of which are in agreement with the records of the Society. In our opinion, these statements, with notes thereto, present fairly the financial position of the Society at December 31, 1946 and the results of its operations for the year ended that date.

Respectfully submitted,
JOHN HEINS & CO.
(Signed) I. RUSSELL BUSH,
Certified Public Accountant.

BALANCE SHEET AS OF DECEMBER 31, 1946

(Including Special and Designated Funds)

ASSETS

General Funds:			
Current: Cash	$70 100.03		
Less check No. 1916 as of December 31, 1946 (estimated cost of 1946 Proceedings and 1946 Book of Standards)	65 000.00		
		$5 100.03	
Investments (Market Value $158 506.64)—Cost		160 383.60	
Accounts Receivable		16 150.27	
Note Receivable (Building Fund)		10 000.00	
Total Current Assets—General Funds		$191 633.90	
Furniture and Fixtures (depreciated book value)		14 964.50	
Total Assets General Funds			$206 598.40
Building Fund:			
Cash		$3 908.47	
Investments (Market Value $25 718.75)—Cost		25 390.63	
Land		30 000.00	
Building		26 503.35	
Construction Costs		102 274.75	
Total Building Fund			$188 077.20

Other Special and Designated Funds:

Cash: A.S.T.M. Research Fund	$6 614.96	
Dudley Medal and Marburg Lecture Fund	253.18	
Richard L. Templin Award Fund	113.46	
Committee Funds	14 305.94	
	$21 287.54	
Investments:		
A.S.T.M. Research Fund (Market Value $38 437.01)—Cost	$41 082.03	
Dudley Medal and Marburg Lecture Fund (Market Value $4 277.50)—Cost	6 625.00	
Richard L. Templin Award Fund (Market Value $1028.75) Cost	1 000.00	
Committee Funds (Market Value $9405.00)—Cost	9 000.00	
	$57 707.03	
Total Other Special and Designated Funds		$78 994.57
Total Assets		$473 670.17

LIABILITIES

General Funds:

Current: Accounts Payable:			
Committee A-1	$202.97		
Committee C-1	75.99		
Advance Collections	6 991.15		
Total Current Liabilities—General Fund		$7 270.11	
Life Membership Fund	3 295.20		
Publication Fund	9 590.95		
Book of Standards Reserve	15 000.00		
Executive Retirement Reserve	10 080.00		
Retirement Fund Reserve	3 651.39		
Reserve for Depreciation of Investments	12 000.00		
Special Award Fund	1 500.00		
Surplus	144 210.75		
		199 328.29	
Total Liabilities General Funds			$206 598.40

Building Fund:

Contributions	154 420.23		
Accumulated Income on Invested Funds	906.77		
Mortgage Payable, dated December 23, 1943, due within 5 years with quarterly installments of $187.50 at 5 per cent $23 875.00 reduced to	22 750.00		
Note payable. General Funds	10 000.00		
Total Building Fund			$188 077.20

Other Special and Designated Funds:

A.S.T.M. Research Fund:			
Principal	$43 335.23		
Income	4 361.76	$47 696.99	
Dudley Medal and Marburg Lecture Fund:			
Principal	6 625.00		
Income	253.18	$6 878.13	
Richard L. Templin Award Fund:			
Principal	1 000.00		
Income	113.46	$1 113.46	
Committee Funds, unexpended balances		23 305.94	
Total Other Special and Designated Funds			$78 994.57
Total Liabilitics and Funds			**$473 670.17**

COMPARISON OF GENERAL FUNDS FOR FISCAL YEARS 1942–1946 INCLUSIVE.

At Close of Fiscal Year	Assets				Liabilities			
	Cash	Investments	Accounts Receivable	Furniture and Fixtures	Accounts Payable	Reserve for Book of Standards	Miscellaneous Funds	Surplus
1942	$14 915.67	$93 281.45	$6 846.09	$6 362.86	$5036.28	$13 890.25	$17 902.43	$84 577.11
1943	29 220.41	111 136.68	14 567.65	6 960.38	5569.02	17 113.98	33 541.26	105 660.86
1944	42 637.45	114 237.61	11 986.97	6 780.66	9009.15	17 113.98	37 913.05	111 606 51
1945	46 058.60	153 718.05	15 033.90	6 641.57	5432.16	15 000.00	35 982.49	165 037.47
1946	5 100.03	160 383.60	26 150.27	14 964.50	7270.11	15 000.00	40 117.54	144 210.75

RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD JANUARY 1, 1946 TO DECEMBER 31, 1946

Cash balance, January 1, 1946			$46 058.60

RECEIPTS

Operating Receipts (Budgeted):

Dues and Entrance Fees:		
Current Dues	$126 486.16	
Past Dues	1 196.35	
Advance Dues	3 231.65	
Income, Life Membership Fund	150.00	
Entrance Fees	5 813.00	
Total Dues		$136 877.16
Sale of Publications:		
Books of Standards (Members addl. parts)	$16 980.00	
1944 Book of Standards	27 294.14	
1945 Supplement Book of Standards	22 549.67	
1946 Book of Standards	191.39	
Methods of Chemical Analysis of Metals	2 317.78	
Compilations of Standards	24 765.97	
Separate Standards	7 856.68	
Selected Standards for Students	2 576.33	
Proceedings	6 422.52	
Spring Meeting Papers	1 361.55	
Bulletin Subscriptions (non-members)	1 017.04	
Special & Miscellaneous Publications	28 897.98	
Total Sale of Publications		$142 231.05
Miscellaneous:		
Advertising, Bulletin	$14 925.30	
Advertising, Index	2 129.60	
Interest and Dividends	6 033.95	
A.S.T.M. Exhibit, Annual Meeting	5 371.20	
Registration and other fees, Annual Meeting	3 721.00	
Committee A-1 for Technical Assistant	412.00	
Miscellaneous	382.16	
Total Miscellaneous Items		$32 975.21
Total Operating Receipts (Budgeted)		$312 083.42

Brought Forward		$312 083.42	$46 058.60
Nonoperating Receipts (Not Budgeted):			
Investments Matured or Sold	$30 274.33		
Excess Remittances	979.68		
Committee C-1 for Technical Assistant	2 000.00		
Executive Retirement Reserve	300.00		
Retirement Fund Reserve	826.39		
Joint A.I.M.E.-A.S.T.M. Publication Fund	5 651.20		
Special Award Fund	1 500.00		
Total Nonoperating Receipts		$41 531.60	
Total Receipts			$353 615.02
Total Receipts for 1946 and Cash Balance, January 1, 1946			$399 673.62

DISBURSEMENTS

Operating Disbursements (Budgeted):			
Publications:			
Year Book	$5 438.96		
1944 Book of Standards	1 329.48		
1946 Book of Standards	45 000.00[a]		
Emergency Alternate Provisions	66.00		
Compilations of Standards	7 500.36		
Separate Standards	4 142.15		
Selected Standards for Students	1 067.46		
Chemical Analysis of Metals	7 696.11		
Proceedings	20 000.00[a]		
Preprints	5 569.40		
Spring Meeting Papers	263.98		
A.S.T.M. Bulletin	14 896.90		
Circulars to Members	2 064.91		
Index to Standards	4 096.37		
Special and Miscellaneous Publications	8 584.45		
Authors' Reprints, Electros, etc. at Cost	802.76		
Total Disbursements, Publications		$128 519.29	
Salaries		122 464.03	
General Office Expenses		28 166.30	
Expenses, Technical and District Committees		8 358.32	
Expenses, Meetings		10 945.63	
American Standards Assn.		1 000.00	
Traveling Expenses, Administrative & Special Committees		4 856.83	
Headquarters Occupancy Expenses		11 217.99	
Furniture and Fixtures		10 602.21	
Principal A.S.T.M. Research Fund		2 900.00	
Dues, Contributions, Miscellaneous		390.36	
Legal Services		1 811.80	
Employees Retirement Fund		8 761.77	
Total Operating Disbursements (Budgeted)		$339 994.53	
Nonoperating Disbursements (Not Budgeted):			
Refund of Excess Remittances	$979.68		
Investments	35 301.53		
Accrued Interest	22.71		
Technical Assistant Committee C-1	2 126.21		
Joint A.I.M.E.-A.S.T.M. Publication Fund	6 148.93		
Loan to Building Fund	10 000 00		
Total Nonoperating Disbursements		$54 579.06	
Total Disbursements			$394 573.59
Cash Balance, December 31, 1946			$5 100.03

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[a] These accounts include $65 000, representing a check drawn against the cost of the 1946 Book of Standards, and the 1946 *Proceedings*, but not actually paid on December 31, 1946.

A.S.T.M. BUILDING FUND

DETAILS OF BUILDING FUND CASH RECEIPTS AND DISBURSEMENTS FOR THE PERIOD JANUARY 1, 1946 TO DECEMBER 31, 1946

Cash Balance January 1, 1946			$85 534.67
Receipts			
Contributions 1946		$9 422.00	
Income from Investments:			
Interest on U. S. Treasury Bonds 2 1/2s 1967–72		649.05	
Proceeds from Sales and Salvage of Equipment Net		942.92	
Loan from A.S.T.M. General Funds		10 000.00	$21 013.97
			$106 548.64
Disbursements			
Architect's Fees	$7 500.00		
General Contractor	78 845.87		
Air Conditioning Contract	7 214.66		
Elevator Contract	7 548.04		
Landscaping	275.00		
Modern Door	334.60		
Plaques	150.00		
Lettering	22.00		
Reduction of Mortgage—4 quarterly payments of $187.50 each	750.00		102 640.17
Cash Balance, December 31, 1946			$3 908.47

A.S.T.M. BUILDING FUND

DETAIL OF ASSETS AND LIABILITIES AS OF DECEMBER 31, 1946

Assets

Cash in Girard Trust Co., Philadelphia, Pa.		$3 908.47	
Investments $25 000 U. S. Treasury Bonds 2½s 1967-72 at $101.16. (Market Value $25 718.75)—Cost		25 390.63	
Property, 1916-18 Race St., Philadelphia, Pa.			
Purchase Price	$53 625.00		
Commission to Agent	2 681.25		
Settlement fees, net	197.10		
	$56 503.35		
Allocated to:			
Land		$30 000.00	
Building		26 503.35	
Construction Costs:			
Architect's fee	$8 700.00		
Rent paid to facilitate possession	110.00		
Zoning fees and permits	17.50		
General Contractor (completed work)	78 845.87		
Air Conditioning installation (completed work)	7 214.66		
Elevator	7 548.04		
Air Conditioning Unit	210.00		
Miscellaneous, doors, landscaping, etc.	781.60		
	103 427.67		
Less, Proceeds from sales and salvage of equipment and rentals	1 152.92	102 274.75	
Total Assets			$188 077.20

Liabilities

Contributions		$154 420.43	
Income on Investments:			
Interest on U. S. Treasury Bonds 2½s 1967-72		906.77	
Mortgage Payable—dated Dec. 23, 1943 due within 5 years with quarterly installments of $187.50 at 5 per cent assumed 1945	23 875 00		
Less, 6 quarterly payments of $187.50 each	1 125.00	22 750.00	
Note payable dated October 19, 1946 to A.S.T.M. General Funds, on demand, with interest at 1 per cent per annum		10 000.00	
			$188 077.20

NOTE.—No provision for depreciation etc. taken into account for the period under review—see comment in letter.

In addition to the General Funds of the Society the Treasurer had on hand the following funds as of December 31, 1946:

COMMITTEE FUNDS

Committee A-1 on Steel	$1 849.72	
Committee A-5 on Corrosion of Iron and Steel	2 960.98	
Committee B-3 on Corrosion of Non-ferrous Metals and Alloys	2 579.44	
Committee B-6 on Die Cast Metals and Alloys	2 513.50	
Committee B-8 on Electrodeposited Metallic Coatings	23.43	
Committee C-1 on Cement	845.76	
Committee C-1 on Master Builders Co. Special Fund	3 666.53	
Committee C-1—Cement Reference Laboratory	484.40	
Committee C-9 on Concrete and Concrete Aggregates	200.82	
Committee C-9 on Sanford E. Thompson Medal Fund	985.25	
Committee C-15 on Manufactured Masonry Units	378.56	
Committee C-18 on Natural Building Stones and Slate	176.36	
Committee D-1 on Paint, Varnish, Lacquer, and Related Products	91.60	
Committee D-2 on Petroleum Products and Lubricants	167.01	
Committee D-5 on Coal and Coke	1 405.24	
Committee D-12 on Soaps and Other Detergents	94.68	
Committee D-13 on Textile Materials	868.38	
Committee D-13 on Research Textile Finishes	238.34	
Committee E-3 on Chemical Analysis of Metals	55.11	
Committee E-9 on Fatigue	197.56	
A.S.A. Sectional Committee on Specifications for Cast Iron Pipe and Special Castings	1 750.52	
Detroit District Committee	1 137.08	
Philadelphia District Committee	132.32	
Pittsburgh District Committee	55.50	
Western New York-Ontario District Committee	447.85	
Total Committee Funds		$23 305.94
Accounted for as follows:		
Cash Balance, December 31, 1946	$14 305.94	
Investments—at cost	9 000.00	
		$23 305.94

A.S.T.M. RESEARCH FUND

	Total	Invested	Uninvested Cash
Principal Account:			
Balance January 1, 1946	$40 281.49	$38 686.24	$1 595.25
Receipts:			
Contribution—A.S.T.M. Current Entrance Fees	+2 900.00		+2 900.00
Sale of Securities		−8 329.05	+8 329.05
Profit on Securities sold	+153.74	+153.74	
	$43 335.23	$30 510.93	$12 824.30
Disbursements:			
Purchase of Securities		+10 571.10	−10 571.10
Balance, December 31, 1946	$43 335.23	$41 082.03	$2 253.20
Income Account:			
Balance January 1, 1946		$3 548.11	
Receipts:			
Interest and Dividends on Investments		1 465.65	$5 013.76
Disbursements:			
Engineering Foundation, Welding Research	250.00		
Committee D-9, Mica Project	102.00		
Committee A-3, Fatigue of Cast Iron	300.00		652.00
Cash Balance December 31, 1946			$4 361.76

DUDLEY MEDAL AND MARBURG LECTURE FUND

Balance January 1, 1946			
Principal—Investments (at cost)	$6 625.00		
Income—Cash	409.43	$7 034.43	
Receipts			
Interest on Investments		86.00	
			$7 120.43
Disbursements			
Honorarium	$200.00		
Cost of Medal	38.50		
Lecture Certificate	3.75		242.25
			$6 878.18
Balance, December 31, 1946:			
Principal—Investments (at cost)		$6 625.00	
Income—Cash		253.18	
			$6 878.18

RICHARD L. TEMPLIN AWARD FUND

Balance, January 1, 1946			
Principal—Investments (at cost)	$1 000.00		
Income—Cash	112.50	$1 112.50	
Receipts			
Interest on Investments		25.96	
			$1 138.46
Disbursements			
Templin Award			25.00
Balance, December 31, 1946			$1 113.46
Principal—Investments (at cost)		$1 000.00	
Income—Cash		113.46	
			$1 113.46

INVESTMENTS

AS OF DECEMBER 31, 1946

GENERAL FUNDS

Par Value		*Cost or Book Value*	*Market Value*[a]
	Bonds		
$2 000	Associated Electric Co., 4½s, due 1953	$1 693.50	$2 050.00
4 000	Erie R. R. Co. General Mortgage Income 4½s, due 2015	2 832.00	3 155.00
2 000	Hudson Coal Co. S.F. 5s Series A, due 1962	1 697.25	1 670.00
3 000	Southern Pacific Deb. 4½s, due 1969	2 544.75	2 910.00
13 000	U. S. Savings Bonds, Series G, 2½s[a]	13 000.00	12 317.00
4 800	U. S. Treasury Bonds, 2½s, due 1967/72	4 800.00	4 938.00
2 000	U. S. Treasury Bonds, 2½s, due 1964/69	2 000.00	2 081.88
2 000	U. S. Treasury Bonds, 2s, due 1952/54	2 000.00	2 057.50
50 000	U. S. Treasury Bonds, 2s, due 1952/54	50 765.63	51 437.50
5 000	Walworth Co. 3¼s Convertible Debentures, due 1976	5 496.53	4 850.00
		$86 829.66	$87 466.88
Shares	*Stock*		
22	American Airlines Inc., 5½ per cent Cumulative convertible, preferred	2 244.00	1 534.50
19	American Cyanamid Co., 5 per cent Cumulative preferred	190.00	209.00
25	Colgate-Palmolive-Peet Co., $3.50 perferred	2 525.00	2 500.00
100	Murray Corp. of America, Cumulative preferred 4 per cent	5 050.06	3 600.00
20	Philco Corp., 3¾ per cent, Series A preferred	2 030.00	1 860.00
75	Allis-Chalmers Mfg. Co., common, no par value	3 316.20	2 718.75
25	American Can Co., common	2 122.30	2 287.50
50	American Cyanamid Co., Class B, common	1 764.25	2 750.00
100	American Rad. & Std. Sanitary Corp. common	1 564.32	1 600.00
25	American Tobacco Co., common	2 110.12	2 025.00
30	American Viscose Corp., common	2 323.58	1 755.00
100	Atlantic Refining Co., common	2 302.50	3 600.00
100	Baldwin Locomotive Works, common	3 822.00	2 075.00
100	Carpenter Steel Co., common	5 426.00	4 900.00
10	Delaware Power & Light Co., common	135.00	230.00
75	General Telephone Corp., common	2 675.00	2 671.88
100	International Harvester, common	9 610.00	7 200.00
60	Lone Star Cement, common, no par value	4 349.42	4 560.00
100	S. S. Kresge Co., common	2 490.18	3 550.00
100	Mathieson Alkali Works, common	3 016.00	2 925.00
75	J. C. Penney Co., common	2 279.63	3 356.25
67	Philadelphia Electric Co., common	1 185.06	1 800.63
116	Public Service Corp. of N. J. common	4 369.50	2 610.00
50	The Texas Company	2 519.55	2 987.50
20	United Gas Improvement Co., common	315.00	465.00
50	U. S. Gypsum, common	3 819.27	5 268.75
		$73 553.94	$71 039.76
	TOTALS	$160 383.60	$158 506.64

A.S.T.M. BUILDING FUND

Par Value			
	Bonds		
$25 000	U. S. Treasury Bonds, 2½s, due 1967–72	$25 390.63	$25 718.75

A.S.T.M. RESEARCH FUND

Par Value		*Cost or Book Value*	*Market Value*[a]
	Bonds		
$2 000	Associated Electric Co. 4½s—1953	1 693.50	2 050.00
1 500	Balto. & Ohio R. R. Gen. & Ref. 5s ser. F. 1996	1 511.25	982.50
2 000	Hudson Coal Co. S. F. 5s Series A, 1962	1 697.25	1 670.00
4 500	U. S. Defense Savings Bonds, Ser. G 2½s[a]	4 500.00	4 263.00
2 000	U. S. Treasury Bonds, 1965/70, 2½s	2 000.00	2 080.00
		$11 402.00	$11 045.50
Shares	***Stock***		
30	Allis-Chalmers Mfg. Co. 3¼ cumulative convertible preferred	3 007.22	2 820.00
33	American Airlines Inc., 3½ cumulative convertible preferred	3 366.00	2 301.75
30	Butler Bros., cumulative preferred	3 090.00	3 255.00
12	Consolidated Edison Co. of N. Y. $5 preferred	1 234.39	1 287.00
29	Consumers Power Co. $4.50 preferred, no par value	2 914.50	3 273.38
20	Philco Corp. 3¾ Series A preferred	2 030.00	1 860.00
100	Allis-Chalmers Mfg. Co., common, no par value	4 421.59	3 625.00
5	Delaware Power & Light Co., common	67.50	115.00
98	General Motors Corp. common, par $10–gift[b]	6 504.75	5 145.00
100	Ohio Oil Co., common	2 167.88	2 387.50
33	Philadelphia Electric Co., common	583.70	886.88
9	Public Service Corp. of N. J., common	135.00	202.50
10	United Gas Improvement Co., common	157.50	232.50
		$29 680.03	$27 391.51
	TOTALS	$41 082.03	$38 437.01

DUDLEY MEDAL AND MARBURG LECTURE FUND

Par Value		*Cost or Book Value*	*Market Value*
	Bonds		
$500.00	Balto. & Ohio R. R. Refunding & General Mortgage 6s, Ser. C 1995	$550.00	$377.50
6 000.00	Balto. & Ohio R. R Refunding & General Mortgage 5s, Ser. A 1995	6 075.00	3 900.00
		$6 625.00	$4 277.50

RICHARD L. TEMPLIN AWARD FUND

$1 000.00	U. S. Treasury Bonds, 2½s, due 1967–72	$1 000.00	$1 028.75

COMMITTEE FUNDS

$1 000.00	U. S. Treasury Bonds, 2⅞s, due 1960–55	$1 000.00	$1 118.12
2 000.00	U. S. Treasury Bons, 2½s, 1964–69	2 000.00	2 080.63
3 000.00	U. S. Treasury Bonds, 2½s, 1965–70	3 000.00	3 120.00
3 000.00	U. S. Treasury Bonds, 2½s, 1967–72	3 000.00	3 086.25
		$9 000.00	$9 405.00
	GRAND TOTAL	$243 481.26	$237 373.65

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[a] Market value is stated at redemption value as of December 31, 1946.

[b] The income from these shares is, by agreement, paid to an annuitant.

APPENDIX II

REPORT OF ADMINISTRATIVE COMMITTEE ON STANDARDS

The Committee on Standards has a very broad assignment as to its functions:

1. To promote the standardization work of the Society,
2. To consider means of general policy concerning standardization activities, including relationship with similar activities of other bodies,
3. To consider the desirability of expansion of standardization work into new fields,
4. To review annually the progress in the Society's standardization work,
5. To pass upon proposed tentatives, tentative revisions of standards, and revisions of tentatives offered between annual meetings of the Society,
6. To pass upon the withdrawal of standards, tentatives, and tentative revisions of standards, on the request of a technical committee between annual meetings of the Society, and
7. To act in an advisory capacity to the Board of Directors in standardization activities in general.

In addition to the functions stated above, the committee has also had the assignment in recent years of acting upon recommendations relating to emergency standards and emergency alternate provisions. The emergency procedure, however, has been discontinued during the year by action of the Board of Directors, and all of the emergency alternate provisions have been discontinued or incorporated in the existing standards or tentatives, and all of the emergency standards have been discontinued with one exception, namely, ES-32, Emergency Method of Test for Color of the U. S. Army Motor Fuel (All Purpose) by Means of ASTM Color Standard, which has been continued at the request of the U. S. Army Ordnance Dept. since Army specifications still carry reference to it.

Much of the committee's attention during the past year has been devoted to routine matters such as reviewing and taking action with respect to recommendations falling under Item 5 above. The number of such recommendations coming to it continues to be large. Two meetings of the committee were held, one on September 9, 1946, and the other on February 24, 1947, at which such recommendations were considered. In addition, however, to the recommendations considered at meetings, quite a number were acted upon by letter. As a result of actions taken, 48 new tentatives were accepted as well as 34 revisions of tentatives, and 13 tentative revisions of standards. In reviewing recommendations of technical committees with respect to standards, the committee is particularly interested in having a representative vote recorded and to be assured that any negative viewpoints have been given adequate consideration. As a matter of record, a list of the recommendations approved appears in the Supplement to this report.

Apart from what might be considered its routine activities, the committee gave consideration to a number of new projects that should be undertaken and is also giving considerable thought to the character and quality of the Society's standards in general. Actions and recommendations in these connections are set forth below.

Expansion of Standardization Activities:

Even though unprecedented expansion has taken place in the past two years, several additional projects are under consideration. Of the projects referred to in the report presented a year ago, organization has been completed of Committee C-17 on Asbestos-Cement Products. An organization meeting is being called of Committee C-2 on Oxychloride-Cement some time in May, and similarly, it is expected that an organization meeting will be held shortly of Committee D-15 on Engine Antifreeze Materials.

Sandwich Materials.—A conference to consider what work should be undertaken in the Society on sandwich type materials was held on June 7, 1946. In light of the discussion at that conference, a special study committee was appointed under the chairmanship of Mr. L. J. Markwardt, and this committee is now reporting to the Board of Directors that a committee on structural sandwich constructions be established within the Society as soon as practicable, to be responsible for problems relating to structural sandwich constructions. So far as possible this committee should call on existing technical committees for test procedures and specifications relating to individual component materials, including adhesives.

Gas Turbines.—A conference was called of those interested in gas turbine materials at which there was considerable interest expressed in having the Society undertake work in this field. The Joint Committee on Effect of Temperature on the Properties of Metals, under the joint auspices of A.S.T.M. and A.S.M.E., may be in a position through suitable reorganization to handle this work, and accordingly this possibility is being explored on the basis of the recommendation of the conference.

Appearance.—The Committee on Standards is now discussing with the ASTM technical committees the need for the coordination of work dealing with the optical properties of materials. A number of committees are now concerned with certain phases of this subject and a separate committee might serve a very useful purpose, both by way of coordinating the activities that are now under way in the individual committees, and also through the pointing up of fundamental aspects of this subject. There is some thought that this committee should include all determinations of sensory perception such as odor and taste, but this question needs to be resolved.

Acoustical Materials.—The Society has maintained liaison representation on the Sectional Committee Z24 on Acoustical Measurements and Terminology since a number of A.S.T.M. committees are interested in this subject. Sound absorption is assuming greater importance in industry in connection with building materials and increasingly in the automotive, aircraft, and railroad fields. In view of this, the Standards Committee recommends that a conference be held of those interested, looking toward the organization of a committee to deal with acoustical materials.

Quality and Form of Standards:

While under the promotion of the standardization work of the Society the expansion of standardization activities is primarily contemplated, the Standards Committee is just as interested in improving the quality of A.S.T.M. standards. It is now discussing with the technical committees ways in which the quality might be improved from the editorial and technical points of view. Some of the technical committees give a great deal of attention to this phase of its work and have organized special editorial committees. The Standards Committee would wish to see all of the A.S.T.M.

standards of a universally high level of quality.

Relations with American Standards Association:

Two methods under the procedure of the American Standards Association are used by A.S.T.M. in submitting recommendations to the ASA. One is a combination of the so called existing standards procedure and the proprietary sponsorship procedure. On the initial reference of an existing A.S.T.M. standard to the ASA, it is submitted under the existing standards procedure with a supporting statement indicating the degree of acceptance. If approved, the A.S.T.M. is granted proprietary sponsorship so far as any future revisions are concerned and such revisions are brought to the attention of the ASA currently for approval under this procedure.

The second method consists of having standards reviewed in a sectional committee made up of representatives of a number of interested organizations, but with A.S.T.M. as sponsor or co-sponsor for the sectional committee. No new standards have been submitted during the past year under the existing standards procedure, but a number of revisions have been submitted with A.S.T.M. as proprietary sponsor as follows:

Revised Standards Submitted Under Proprietary Procedure.—The Society has been granted proprietary sponsorship for the revision of A.S.T.M. standards approved as American Standard under the existing standards procedure and, functioning as proprietary sponsor, has submitted to the American Standards Association during the year revisions of the following standards:

Standard Specifications for:

Gray Iron Castings (ASTM A 48 - 46; ASA G25.1—1947),
Slab Zinc (Spelter) (ASTM B 6 - 46; ASA H24.1—1946),
Free-Cutting Brass Rod and Bar for Use in Screw Machines (ASTM B 16 - 46; ASA H8.1—1946),
Copper Water Tube (ASTM B 88 - 46; ASA H23.1—1946),
Copper and Copper-Base Alloy Forging Rods, Bars and Shapes (ASTM B 124 - 46; ASA H7.1—1946),
Lampblack (ASTM D 209 - 46; ASA K26.1—1946),
Bone Black (ASTM D 210 - 46; ASA K36.1—1946),
Prussian Blue (ASTM D 261 - 46; ASA K29.1—1946), and
Chromium Oxide Green (ASTM D 263 - 46; ASA K 37.1—1946).

Standard Methods of:

Laboratory Sampling and Analysis of Coal (ASTM D 271 - 46; ASA K18.1—1946).

Standards Submitted Under Sectional Committee Procedure.—Reports were received from five of the sectional committees for which the Society is sponsor or joint sponsor, submitting standards to the ASA for approval. These reports were transmitted to that association and the following standards have been approved for the first time or reapproved in revised form during the year as American Standard.

On the Recommendation of Sectional Committee A37 on Road and Paving Materials.

Standard Methods of Test for:

Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (ASTM C 131 - 46; ASA A37.7—1947), and
Sieve Analysis of Fine and Coarse Aggregates (ASTM C 136 - 46; ASA A37.8—1947).

On the Recommendation of Sectional Committee A42 on Specifications for Plastering

Standard Specifications for:

Gypsum Plastering Including Requirements for Lathing and Furring (A42.1—1946),
Portland Cement Stucco (A42.2—1946), and
Portland Cement Plastering (A42.3—1946).

On the Recommendation of Sectional Committee H4 on Copper Wire

Standard Specifications for:

Soft or Annealed Copper Wire (B 3 - 46; ASA H4.1—1947),
Bronze Trolley Wire (ASTM B 9 - 46; ASA H4.5—1947),
Tinned Soft or Annealed Copper Wire for Electrical Purposes (ASTM B 33 - 46; ASA H4.4—1947) and
Copper Trolley Wire (ASTM B 47 - 46; ASA H4.6—1947).

On the Recommendation of Sectional Committee C59 on Electrical Insulating Materials

Tentative Methods of Testing Molded Materials Used for Electrical Insulation ASTM D 48 - 46 T; ASA C59.1—1947).

On the Recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants

Standard Methods of Test for:

Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (ASTM D 86 - 46; ASA Z11.10—1947),
Flash and Fire Points by Means of Open Cup (ASTM D 92 - 46; ASA Z11.6—1947),
Flash Point by Means of the Pensky-Martens Closed Tester (ASTM D 93 - 46; ASA Z11.7—1947),
Water in Petroleum Products and Other Bituminous Materials (ASTM D 95 - 46; ASA Z11.9—1947),
Water and Sediment in Petroleum Products by Means of Centrifuge (ASTM D 96 - 46; ASA Z11.8—1947),
Carbon Residue of Petroleum Products (Conradson Carbon Residue) (ASTM D 189 - 46; ASA Z11.25—1947),
Knock Characteristics of Motor Fuels (ASTM D 357 - 46; ASA Z11.37—1947),
Gum Content of Gasoline (ASTM D 381 - 46; ASA Z11.36—1947), and
Ash Content of Petroleum Oils (ASTM D 482 - 46; ASA Z11.54—1947).

In accordance with ASA procedure, the association has, on the recommendation of the Society, reaffirmed a number of A.S.T.M. standards which had been approved as American Standard and have stood over three years without revision.

International Organization for Standardization:

In the report submitted a year ago, reference was made to the United Nations Standards Coordinating Committee and certain projects that were under consideration for international discussion under the jurisdiction of that organization. Since then, steps have been taken to organize a new International Organization for Standardization which will supersede the old International Standards Association as well as the UNSCC. A charter and by-laws have been drafted of which the American Standards Association has already indicated its approval and thereby becomes a member of this international organization. The A.S.T.M. is cooperating with the ASA in considering various proposals for international projects, since many of these will deal directly with materials. At the moment, informal discussions have taken place looking toward the revival of a number of the old I.S.A. projects, including the one on methods of testing petroleum products for which the United States held the secretariat. Several projects are now officially under consideration and A.S.T.M. has been asked to express an opinion regarding the desirability of establishing formal projects in these fields together with recommendations with respect to scope.

Viscosity.—There is some thought that this project should be limited to reference standards for absolute viscosity and apparatus for measuring absolute viscosity.

Plastics.—It is understood that this relates primarily to methods of testing and nomenclature of plastics.

Textiles.—Sectional Committee L14 on Textile Test Methods is suggesting that methods of testing textiles be set up as a separate project in line with the proposal submitted to the UNSCC a year ago and that the United States be given the secretariat for this project.

Other International Relations:

Apart from the international projects being set up under the I.S.O., the Society has maintained exchange relations with a large number of the standardization organizations throughout the world. A more intimate relationship has been established with Great Britain and comments have been solicited from A.S.T.M. committees with respect to a large number of draft standards submitted to us by the British Standards Institution. Comments have been transmitted to the B.S.I. and have been deeply appreciated.

A similar arrangement has been suggested for exchange of comments with the Chinese Standards Association and the Argentina Standards Association (I.R.A.M.).

Respectfully submitted on behalf of the committee,

R. D. BONNEY,
Chairman.

R. E. HESS,
Secretary.

SUPPLEMENT

The following new specifications, methods of test, definitions, and recommended practices were accepted by the Standards Committee during the year for publication as tentative:

Tentative Specifications for:

Carbon-Steel and Alloy-Steel Forgings for Magnetic Retaining Rings for Turbine Generators (A 288—46 T),
Alloy-Steel Forgings for Nonmagnetic Coil Retaining Rings for Turbine Generators (A 289 – 46 T),
Carbon-Steel Forgings for Rings for Main Reduction Gears (A 290 – 46 T),
Carbon-Steel and Alloy-Steel Forgings for Pinions for Main Reduction Gears (A 291 – 46 T),
Carbon-Steel and Alloy-Steel Forgings for Turbine Generator Rotors and Shafts (A 292 – 46 T),
Carbon-Steel and Alloy-Steel Forgings for Turbine Rotors and Shafts (A 293 – 46 T),
Carbon Steel and Alloy-Steel Forgings for Turbine Bucket Wheels (A 294 – 46 T),
Corrosion-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 – 46 T),
Heat-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 297 – 46 T),
Corrosion-Resisting Chromium and Chromium-Nickel Steel Welding Electrodes (A 298 – 46 T),
White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (B 23 – 46 T),
Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 46 T),
Fire-Refined Copper for Wrought Alloys (B 216 – 46 T),
Magnesium-Base Alloy Extruded Round Tubing (B 217 – 46 T), and
Structural Clay Facing Tile (C 212 – 46 T).

Tentative Methods of:

Testing Electrical Insulating Oils (D 117 – 46 T),
Test for Changes in Protective Properties of Coatings of Paint, Varnish, Lacquer, and Related Products on Steel Surfaces When Subjected to Immersion (D 870 – 46 T),
Test for Foaming Characteristics of Crankcase Oils (D 892 – 46 T),
Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils (D 893 – 46 T),
Test for Sulfur in Lubricating Oils Containing Additives and in Additive Concentrates by Bomb Method (D 894 – 46 T),
Test for Resistance of Adhesive Bonds to Chemical Reagents (D 896 – 46 T),
Test for Tensile Properties of Adhesives (D 897 – 46 T),
Test for Determining the Applied Weight Per Unit Area of Dried Adhesive Solids (D 898 – 47 T),
Test for Determining the Applied Weight Per Unit Area of Liquid Adhesives (D 899 – 47 T),
Test for Calorific Value of Gaseous Fuels by the Water-Flow Calorimeter (D 900 – 46 T),
Testing Askarels (D 901 – 46 T),
Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used in Electrical Insulation (D 902 – 46 T),
Test for Peel or Stripping Strength of Adhesives (D 903 – 46 T),
Testing the Strength Properties of Adhesives in Shear by Compression Loading (D 905 – 47 T),
Testing the Strength Properties of Adhesives in Plywood Type Construction in Shear by Tension Loading (D 906 – 47 T),
Chemical Analysis for Sulfur by Direct Combustion and for Nitrogen of Steel (E 30 – 46 T),

Chemical Analysis of Ferro-Alloys (E 31 – 46 T),
Polarographic Determination of Lead and Cadmium in Slab Zinc (Spelter) (E 40 – 46 T),
Chemical Analysis of Special Brasses and Bronzes (E 54 – 46 T),
Sampling Wrought Non-Ferrous Metals and Alloys for Determination of Chemical Composition (E 55 – 46 T),
Chemical Analysis of White Metal Bearing Alloys (E 57 – 46 T),
Chemical Analysis of Magnesium and Magnesium-Base Alloys (E 61 – 46 T),
Chemical Analysis of Copper and Copper-Base Alloys (E 62 – 46 T),
Determination of Iron in (70–30) Copper-Nickel Alloy (E 63 – 46 T),
Determination of Iron in Slab Zinc (Spelter) (E 64 – 46 T),
Sampling Slab Zinc (Spelter) (E 65 – 46 T),
Determination of Iron in Lead- and Tin-Base Alloys (E 67 – 46 T),
Determination of the pH of Aqueous Solutions with the Glass Electrode (E 70 – 46 T), and
Industrial Radiographic Standards for Steel Castings (E 71 – 47 T).

Tentative Definitions of:

Terms Relating to Structural Clay Tile (C 43 – 46 T), and
Terms Relating to Plastics (D 883 – 46 T).

Tentative Recommended Practices for:

Determining the Effect of Artificial (Carbon-Arc Type) and Natural Light on the Permanence of Adhesives (D 904 – 46 T), and
Photometric Methods for Chemical Analysis of Metals (E 60 – 46 T).

Revisions of the following tentatives and tentative revisions of standards were accepted by the Standards Committee during the year:

Tentative Specifications for:

Light Gage Structural Quality Flat Hot-Rolled Carbon Steel (0.2499 and 0.1874 in. to 0.0478 in. in Thickness) (A 245 – 47 T),
Light Gage Structural Quality Flat Rolled Carbon Steel (0.0477 to 0.0225 in. in Thickness) (A 246 – 47 T),
Iron and Steel Gas-Welding Rods (A 251 – 46 T),
Carbon-Chromium Ball and Roller-Bearing Steels (A 295 - 46 T),
Aluminum-Base Alloy Sand Castings (B 26 – 46 T),
Soft Solder Metal (B 32 – 46 T),
Aluminum for Use in Iron and Steel Manufacture (B 37 – 46 T),
Magnesium-Base Alloy Sheet (B 90 – 46 T),
Aluminum-Base Alloy Permanent Mold Castings (B 108 – 46 T),
Aluminum and Aluminum-Alloy Sheets and Plates (B 209 – 46 T),
Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 – 46 T),
Rubber Sheath Compound for Electrical Insulated Cords and Cables (D 532 – 46 T),
Insulated Wire and Cable: Ozone-Resistant Type Insulation (D 574 – 46 T),
Cellulose Acetate Molding Compounds (D 706 – 46 T),
Laminated Thermosetting Materials (D 709 – 46 T),
Insulated Wire and Cable: Heat-Resisting Synthetic Rubber Compound (D 754 – 46 T),
Insulated Wire and Cable: Performance Synthetic Rubber Compound (D 755 – 46 T),
Cellulose Acetate Plastic Sheets (D 786 – 46 T),
Ethyl Cellulose Molding Compound (D 787 – 46 T), and
A.S.T.M. Thermometers (E 1 – 46 T).

Tentative Methods of:

End-Quench Test for Hardenability of Steel (A 255 – 46 T),
Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 – 46 T),
Testing Rubber Hose (D 380 – 46 T),
Testing Rubber Insulated Wire and Cable (D 470 – 46 T),
Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 609 – 46 T),
Testing Vulcanized Fibre Used for Electrical Insulation (D 619 - 46 T),
Testing Rubber-Coated Fabrics (D 751 – 46 T),
Test for Shipping Containers in Revolving Hexagonal Drum (D 782 – 46 T),
Identification and Quantitative Analysis of Synthetic Elastomers (D 833 – 46 T),
Test for Evaluating Degree of Settling of Traffic Paint (D 869 – 46 T),
Compression Testing of Metallic Materials (E 9 – 46 T), and
Chemical Analysis of Lead- and Tin-Base Solder Metal (E 46 – 46 T).

Tentative Recommended Practice for:

Operating Light and Water Exposure Apparatus (Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products (D 822 – 46 T), and
Apparatus and Reagents for Chemical Analysis of Metals (E 50 – 46 T).

Tentative Revision of Standard Specifications for:

Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 44),

Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 - 44),
Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 44),
Welded Alloy-Steel Boiler and Superheater Tubes (A 249 - 44),
Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 - 44),
Structural Clay Load-Bearing Wall Tile (C 34 - 41),
Structural Clay Non-Load Bearing Tile (C 56 - 41),
Structural Clay Floor Tile (C 57 - 39),
Building Brick (Made from Clay or Shale) (C 62 - 44), and
C. P. Zinc Yellow (Zinc Chromate) (D 478 - 41).

Tentative Revision of Standard Methods of:

Sampling and Testing Brick (C 67 - 44),
Test for Compressive Strength of Hydraulic Cement Mortars (C 109 - 44), and
Sampling and Testing Structural Clay Tile (C 112 - 36).

APPENDIX III

REPORT OF ADMINISTRATIVE COMMITTEE ON RESEARCH

The Committee has held two meetings at Society Headquarters: the first a two-day meeting on October 17 and 18, 1946, and the second on February 24, 1947.

The meeting on October 17 concerned itself with a review of all of the research activities being carried on by committees of the Society. A questionnaire had been sent out asking the officers of the committees to list all such activities, and in addition representatives of the Secretary's office had contacted committees which had not replied to the questionnaire so that a rather complete report of all committee activities was available. While there was some discussion of definitions of such terms as "pure research," and "applied research," an attempt was made to appraise the various activities of the committees as research or as testing for the purpose of preparation of specifications. It was realized that this distinction could not be made absolute. It was the purpose of this survey to classify A.S.T.M. activities so as to know what subjects were of special interest to the committee, and to select subjects to be included in a Review of A.S.T.M. Research Projects to be published in the ASTM BULLETIN.

On October 18, the committee met with the Administrative Committee on Simulated Service Testing. This served to acquaint the Committee on Research with the activities of this recently organized committee with a view to correlating, in so far as possible, the activities of the two committees.

A feature of the February 24 meeting was the conference with officers of selected A.S.T.M. committees. These included Committees A-9 on Ferroalloys, C-16 on Thermal Insulating Materials, D-3 on Gaseous Fuels, D-14 on Adhesives, and D-20 on Plastics.

Some of these committees have within their own organization a committee on research or its equivalent, and they have worked out comprehensive research campaigns. The Administrative Committee on Research is considering ways and means of encouraging this type of activity in other committees, especially in fields where it seems possible to conduct research work in a cooperative way or even to sponsor research work in some specially qualified laboratory or organization.

The Administrative Committee on Research has assisted certain committees by contributions or by fund transfer during the year as follows:

Committee A-3 on Cast Iron:	
Testing of cast iron at elevated temperatures	$300
Administrative Committee on Ultimate Consumer Goods:	
For preliminary studies on procedures for establishment of test methods and specifications for consumer goods	$600
Advisory Committee on Corrosion:	
A loan to meet costs of test racks for Committee B-8 corrosion research	$500

In addition to attempting to keep in touch with the research activities of the various A.S.T.M. committees, some attention has been given to the research activities of other organizations as reported by the National Research Council and Government agencies. The objective has been to see what could be done in the way of correlation.

Current Problems and Future Work:

The committee has for consideration the problem of stimulating research in various committees of the Society by encouraging the formation of research subcommittees in the various committees, and by correlating the activities of various committees. Ways and means of making more extensive use of the funds available from the research fund are being studied with the full realization that the funds are somewhat limited. Consideration is also being given to increasing the research fund.

The committee has drawn up a form of "Application for Grant of A.S.T.M. Research Funds" which it is believed will facilitate the consideration by this committee of requests for financial aid for A.S.T.M. committee research projects.

A major problem which has continued to face the committee is how to function with so many problems and such limited funds. (At present the annual income from the principal of the Research Fund is only about $1200.) In the past, the funds have been used almost entirely for emergency purposes; that is, a research of value which is almost completed, and a small contribution from the research fund would provide for its completion. This policy can be continued and may be the best procedure. However, the activities of the Research Committee might be extended substantially and to advantage if more funds were made available. It is not even suggested that the Research Committee undertake an extensive campaign of sponsoring researches. Many of the individual committees are able to organize, finance, and conduct the researches needed in their particular fields. However, it is believed that in some cases needed research could be initiated by suggestions and some financial assistance from the Research Committee.

The committee records the loss of a most valuable member and its chairman, Dr. Robert J. Moore who died January 6, 1947. Dr. Oscar E. Harder, Assistant Director, Battelle Memorial Institute, has been elected as chairman for the term ending June, 1948. Dr. P. D. Brossman of E. I. du Pont de Nemours and Company, Inc., has been appointed to membership to June, 1950.

Respectfully submitted on behalf of the committee,

OSCAR E. HARDER,
Chairman.

C. L. WARWICK,
Secretary.

APPENDIX IV

REPORT OF ADMINISTRATIVE COMMITTEE ON PAPERS AND PUBLICATIONS

The Report of the Administrative Committee on Papers and Publications is necessarily largely one of record, presenting as it does the various regular and special publications that have been issued during the past year and indicating those that are in prospect.

The committee is engaged in an extensive study of the entire publication setup and has two committees actively at work. One committee, under the chairmanship of Mr. J. C. Geniesse, is examining the question as to how the standards might best be published. With the continued and rapid growth in the number of standards and tentatives, there is serious question how much longer the present methods of publication will be either physically possible, or desirable. The 1946 edition of the Book of A.S.T.M. Standards is issued in five parts and in accordance with the present plans there would be no further complete reprinting for a three-year interval, but supplements would be issued annually for each of the parts, which would mean that at the end of this period 16 volumes would be involved including the Methods of Chemical Analysis of Metals. Various suggestions that have been made from time to time for modification of the present scheme of publication are being considered, including publication in loose-leaf or loose-unit form, or publication in the form of a number of separate books covering the various groups of materials, much along the lines of the present "compilations" that have proved so useful and popular.

A second committee, under the chairmanship of Mr. P. G. McVetty, is making a thorough and indeed fundamental study of the publication of the *Proceedings*, special technical publications, and ASTM BULLETIN. It will be recalled that two years ago there were instituted publication procedures designed to encourage the offering of papers at any time throughout the year and preprinting papers as soon as they become available. Preprints could be either published separately or as part of the ASTM BULLETIN; in the first instances synopses together with announcements and order sheets are published in the BULLETIN. It was also decided to continue to publish in the BULLETIN those technical papers, articles, and committee reports which appear to have current technical or educational value but which do not seem to warrant separate publication or inclusion in a bound volume. Only a limited degree of success has thus far been attained in spreading out over the year the publication of technical papers, but to a greater extent than ever technical papers and articles not intended for *Proceedings* are appearing in the BULLETIN. At least two schools of thought exist in the Society today: One is that the publication of technical papers should be on the basis of quarterly or even monthly *Proceedings* instead of in the annual bound volume. Some who hold this view feel that the technical papers should be in quarterly or monthly *Proceedings* and not in the BULLETIN. The other school of thought is that the BULLETIN should be expanded into a technical journal which would contain as a separate section the technical papers of the Society and that the BULLETIN should be issued more than six times a year, possibly ten times or even monthly. Some members hold views that are intermediate between these extremes. It is the task of this second committee to study these and other plans of publication and to determine as well as possible in the light of present problems and future developments what is thought to be the most desirable publication plan, taking into account also the proper place of the numerous special publications that the Society now issues.

The committee is examining the publication policies of a number of our sister technical societies, some of whom have apparently been confronted with problems similar to ours and have recently made extensive changes in the methods of issuing their publications. The determination of the most desirable publication policy involves a number of considerations, such as page size, format, desirable content of publications, advertising, policy of distribution to members, and the like. It may well be that before a report can be made to the Board of Directors by June of 1948, as the Board directed, the members of the Society should be canvassed for expression of opinion concerning these matters. If so, the Administrative Committee believes that such a canvass would need to be based on a carefully worded questionnaire and should preferably give the members several alternate publication plans from which to choose the one that would seem to be best suited to his particular requirements.

Special Publication Series.—For some time, the Committee on Papers and Publications has had under consideration the assigning of serial numbers to the special publications. Such publications have, of course, been recorded annually in the Report of the Committee on Papers and Publications and attention has been called to them in the List of Publications that has been sent annually to all members. Establishing a special publication series, however, will place these special publications on a more formal basis and should facilitate reference to them. A complete record has been made of all such publications from the time of the incorporation of the Society and serial numbers have been assigned. This list is included in the May issue of the ASTM BULLETIN.

RECORD OF PUBLICATIONS ISSUED THIS YEAR

1946 *Proceedings*, 1614 pp. (estimated), 6900 copies. Available in May, 1947
1946 Book of Standards
  Part I-A, 1211 pp., 12250 copies
  Part I-B, 949 pp., 12000 copies
  Part II, 1810 pp., 10000 copies
  Part III-A, 1322 pp., 9100 copies
  Part III-B, 1402 pp., 9000 copies
Index to 1946 Standards and Tentative Standards, 240 pp. (estimated), 18,000 copies
Year Book, 512 pp., 5300 copies
ASTM BULLETIN, six issues, 612 pp., average edition, 8825

A statement of the volume of the principal publications for the past four years follows:

Publications	1943, pages	1944, pages	1945, pages	1946, pages
Proceedings	1357	1139	1060	1614
Book of A.S.T.M. Standards	....	6042	....	6694
Supplements to Book of A.S.T.M. Standards	1124	....	1189	....
A.S.T.M. Methods of Chemical Analysis of Metals	334	32	....	412
Index to A.S.T.M. Standards and Tentative Standards	218	218	235	240
Year Book	374	408	474	512
Spring Meeting Symposiums	135	140	....	166
ASTM Bulletin	468	464	492	600
	4007	8443	3450	10238

Special Compilations of Standards Published from April 15, 1946 to April 15, 1947:

A.S.T.M. Specifications for Steel Piping Materials (A-1), 306 pp., 2000 copies

A.S.T.M. Standards on Electrical-Heating and Resistance Alloys (B-4), 183 pp., 900 copies

A.S.T.M. Standards on Copper and Copper Alloys (B-5), 477 pp., 1000 copies

Specifications and Tests for Electrodeposited Metallic Coatings (B-8), 52 pp., 5000 copies

A.S.T.M. Standards on Cement (C-1), 192 pp., 2500 copies

A.S.T.M. Standards on Concrete and Concrete Aggregates (C-9), 200 pp., 1500 copies

A.S.T.M. Standards on Glass and Glass Products (C-14), 96 pp., 1000 copies

A.S.T.M. Standards on Petroleum Products and Lubricants (D-2), 627 pp., 6000 copies

A.S.T.M. Standards on Paper and Paper Products (D-6), 240 pp., 1000 copies

A.S.T.M. Standards on Rubber Products (D-11), 551 pp., 1500 copies

A.S.T.M. Standards on Textile Materials (D-13), 504 pp., 2000 copies

Standard Specifications for Gypsum Plastering, 16 pp., 25000 copies

Standard Specifications for Portland Cement Stucco and Portland Cement Plastering, 24 pp., 3000 copies

Special Publications Also Issued:

Selected A.S.T.M. Standards for Students in Engineering, 274 pp., 5000 copies—published in June; 5000 copies photo offset in December

1946 Edgar Marburg Lecture—Protective Organic Coatings as Engineering Materials by J. J. Mattiello, 100 pp., 3500 copies

1945 Supplement to the Bibliography and Abstracts on Electrical Contacts, 20 pp., 1000 copies

Index to the Literature on Spectrochemical Analysis, 184 pp , 2000 copies

Symposium on Adhesives, 68 pp., 2000 copies

Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals, 151 pp., 3000 copies

Discussion on Statistical Quality Control in Its Application to Specification Requirements, 15 pp., 4000 copies

Symposium on Bearings, 71 pp., 3000 copies

Symposium on Materials for Gas Turbines, 205 pp., 2500 copies

Symposium on Oil Procurement Practices, 64 pp., 4000 copies

Symposium on New Methods for Particle Size Determination, 117 pp., 750 copies (reprint)

Forum on Diesel Fuel Oils, 48 pp., 4000 copies—Serial number 71

Special Publications in Prospect:

Symposium on Effects of Low Temperatures on Materials—sponsored by the Philadelphia District Committee at a meeting held in Philadelphia, March, 1946. 100 pp., 4000 copies

Symposium on Ultra-High Voltage and High-Speed Radiography—sponsored by Committee E-7 on Radiography and held in conjunction with the Fifth Annual Convention of The American Industrial Radium and X-Ray Society, Inc., in Cleveland, February, 1946, 150 pp., 3000 copies

Symposium on Testing of Parts and Assemblies—sponsored by the Administrative Committee on Simulated Service Testing and the Society for Experimental Stress Analysis and held at the Annual Meeting of the Society in June, 1946, 98 pp., 3000 copies

Symposium on Spectroscopic Light Sources—sponsored by Committee E-2 on Spectrographic Analysis and held at the Annual Meeting of the Society in June. 1946, 120 pp., 2000 copies

1946 Supplement to Bibliography and Abstracts on Electrical Contacts, 20 pp., 1000 copies

Report on Standard Samples for Spectrochemical Analysis, 32 pp., 2500 copies

Tables of Data on Corrosion-Resistant Steels (A-10), 32 pp., 3200 copies

Symposium on pH Measurement—sponsored by Committee E-1 on Methods of Testing and held at the Annual Meeting of the Society in June, 1946, 80 pp., 2000 copies

In addition to these, the following symposiums are being reprinted from the 1946 *Proceedings*:

Symposium on Freezing-and-Thawing Tests of Concrete, 60 pp., 2000 copies

Symposium on Atmospheric Weathering of Corrosion-Resistant Steels, 91 pp., 2000 copies

Compilations of Standards in Prospect:

Supplement to the Joint Committee Report on Standard Specifications for Concrete and Reinforced Concrete, 130 pp., 1500 copies

A.S.T.M. Standards on Coal and Coke (D-5), 170 pp., 1400 copies

A.S.T.M. Standards on Electrical Insulating Materials (D-9), 600 pp., 1400 copies

Procedures for Testing Soils (D-18), 200 pp., 1800 copies

Current Activities:

Program for 1947 Annual Meeting.—A very full program is in prospect for the 1947 Annual Meeting with a number of symposiums and round-table discussions scheduled for presentation as follows:

Round-Table Discussion on Water-Formed Deposits—under the auspices of Committee D-19 on Industrial Waters,

Discussion on Exposure Test Panels—under the auspices of Committee B 8 on Electro-deposited Metallic Coatings,
Symposium on Synthetic Lubricants—under the auspices of Committee D-2 on Petroleum Products and Lubricants,
Symposium on Insulating Oil—under the auspices of Committee D-9 on Electrical Insulating Materials,
Symposium on Measurement of Entrained Air in Concrete—under the auspices of Committee C-9 on Concrete and Concrete Aggregates,
Symposium on Load Tests on Bearing Capacity of Soils—under the auspices of Committee D-18 on Soils for Engineering Purposes,
Session on Soils (General Interest Papers)—under the auspices of Committee D-18,
Symposium on Rubber Testing—under the auspices of Committee D-11 on Rubber and Rubber-Like Materials, and
Round-Table Discussion on Speed of Testing—under the auspices of Committee E-1 on Methods of Testing.

In addition to the above there are a number of other technical papers not a part of any formal symposium. A number of papers are also being offered to the Society for publication without necessarily having these presented at a meeting of the Society. While these for the most part are published in the ASTM BULLETIN, an increasing number are being preprinted separately and subsequently published in the *Proceedings*.

Respectfully submitted on behalf of the committee,

C. L. WARWICK,
Chairman.

APPENDIX V

REPORT OF ADMINISTRATIVE COMMITTEE ON ULTIMATE CONSUMER GOODS

The Administrative Committee on Ultimate Consumer Goods has held two all-day meetings since the last report, one on June 24, 1946, at Buffalo, N. Y., the other on November 26, 1946, at Philadelphia, Pa. At the latter meeting the committee was pleased to welcome George N. Thompson, Chief, Division on Codes and Specifications, National Bureau of Standards, as a new member.

During the past year the committee has completed the Suggested Method of Test Form and the Suggested Specification Form, both applicable to ultimate consumer goods, referred to in the last report. Copies were sent to the chairmen and secretaries of all technical committees for their information, comment, or criticism.

In order further to publicize the interest of the American Society for Testing Materials in the field of consumer goods, an appropriate article, embodying the Suggested Forms, was prepared by the chairman of the Administrative Committee and published in the October, 1946, issue of the ASTM BULLETIN under the title "Progress in Work on Ultimate Consumer Goods." Many reprints have already been distributed to individuals and organizations actively engaged in work in this field. Copies were also sent to the officers of all technical committees with an inquiry as to whether there were any materials under their respective jurisdictions on which consumer goods standards should be written. Those committees which indicated full or partial interest were B-6, D-8, D-9, D-13, D-20, and E-11.

The Administrative Committee has continued its study of the various factors entering into the development of test methods for ultimate consumer goods and has taken cognizance of the interaction of the physical and social sciences on the subject. Under the guidance of A. G. Ashcroft, the committee has organized two task groups to assist it in formulating a suitable program. Task Group No. 2, under the chairmanship of Prof. C. West Churchman, of the University of Pennsylvania, is working in the area included in "Type 3 Quality"—that which makes a thing wantable in terms of characteristics that make it satisfactory, adequate, dependable, or economic to some one or more persons. Task Group No. 1, under Prof. J. C. Whitwell, of Princeton University, is covering the area of how to set up a standard on "Type I Quality"—that which characterizes a thing itself independent of all other things and of human volition and interests. It is planned to present research programs emanating from the two task groups to the joint NRC-SSRC Committee on Measurement of Opinion, Attitudes, and Consumer Wants through C. L. Warwick, the A.S.T.M. representative. Approval by the joint committee would undoubtedly mean the incorporation of the task group programs into the long-term program of research which the joint committee is planning.

Another problem still under current consideration is what procedure to recommend to technical committees for use in developing standards for consumer goods. The various facets of this problem were fully set forth in the previous annual report and will not be repeated here. However, one statement in that report might bear reiteration. It is the one which expresses agreement of the Administrative Committee that A.S.T.M. standards on consumer goods will not carry proper weight if in their establishment there has not been adequate representation from those groups or organizations which are competent to express the viewpoint of the ultimate consumer.

Respectfully submitted on behalf of the committee,

HERBERT J. BALL,
Chairman.

R. E. HESS,
Secretary.

APPENDIX VI

REPORT OF ADMINISTRATIVE COMMITTEE ON SIMULATED SERVICE TESTING

The Administrative Committee On Simulated Service Testing held one meeting at Society Headquarters during the year on October 18, 1946. This meeting was held in conjunction with a meeting of the Administrative Committee on Research with a view to correlating in so far as possible the work of the two committees, and in forming the latter committee of the current activities in the field of simulated service testing.

The recommendations which were made by the committee last year and approved by the Board of Directors are already beginning to bear fruit. Committee E-9 on Fatigue was organized during the 1946 Annual Meeting of the Society in Buffalo, and the former Research Committee on Fatigue of Metals was reorganized as the research subcommittee of E-9. Committee E-9, including the research subcommittee, is engaged in an active program of work as is separately reported by Committee E-9.

Committee E-6 on Methods of Testing Building Constructions, organized in January, 1946 is energetically attacking various problems in this field. A new Method for Conducting Strength Tests of Panels for Building Constructions was developed by the committee and accepted as tentative by the Administrative Committee on Standards, on May 7, 1947.

The Administrative Committee on Simulated Service Testing was responsible for five well-attended and highly successful technical sessions at the 1946 Annual Meeting in Buffalo. Two of these comprised a Symposium on Fatigue sponsored jointly with Committee E-9 at which eight papers were presented. Two of the sessions comprised a Symposium on Testing of Parts and Assemblies[1] which was jointly sponsored with the Society for Experimental Stress Analysis. Seven papers were presented, most of which emphasized the fatigue testing of finished parts. The fifth session was a Symposium on Bearings,[2] the first such symposium to have been presented before any technical society.

This Bearings Symposium aroused a considerable amount of interest and seems to indicate that continued A.S.T.M. activity in this field may be warranted. To explore this subject further it is planned to form a joint study committee with representatives of A.S.T.M. and representatives of the Annular Bearing Engineers' Committee. The recommendations of this joint study committee should provide the basic information upon which the Committee on Simulated Service Testing can advise the Society as to the position of A.S.T.M. in this field.

The committee has proposed that a symposium be held at the June, 1947, meeting on the subject of metals in the plastic range. Several papers on this subject are in prospect. One will deal with work during the war at the University of California, another with work at the California Institute of Technology, while a third will report on work done at the National Bureau of Standards. A fourth paper of kindred interest will deal with the velocity aspect of tensile-impact testing and treats of work done at the Brooklyn Naval Ship Yard.

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[1] Technical Publication No. 72, Am. Soc. Testing Mats., April, 1947.

[2] Issued as separate publication, January, 1947.

Another subject being actively studied by the committee is the testing of springs, but inquiry has shown that there is not sufficient material available at the present time to hold a symposium on this subject. However, one paper is being contributed by the Ford Motor Co. for the Annual Meeting which will deal with some of the effects of cadmium, zinc, and tin plating on springs. The subject continues on the committee's agenda and it is hoped that by next year more papers will be forthcoming.

The committee received a request from the Ordnance Department for help in the standardization of procedures and equipment for humidity testing with particular reference to simulating tropical conditions. It was decided that this was an appropriate subject for A.S.T.M. activity. The subject was referred for further study to the Technical Committee X on Conditioning and Weathering, of Committee E-1 on Methods of Testing, where the project has now been started.

The committee is also taking an active interest in the work on gas turbine materials which is currently being centered in the Joint Research Committee on Effect of Temperature on the Properties of Metals which sponsored a symposium on the subject at the 1946 Annual Meeting. It is expected that many phases of this problem will deal with the testing of parts and assemblies or simulated service testing.

It is interesting to note that work in simulated service testing has been stimulated in other committees of the Society, notably in Committee D-20 on Plastics, which, after a review of the subject by a special study group, has set up a section on accelerated service testing in its Subcommittee V on Permanence Properties.

The committee is also studying carefully the information developed at a conference and by a study committee on sandwich constructions. It is also reviewing a number of suggested projects, other than humidity, which were presented to it for consideration by the Ordnance Department, as well as several testing techniques developed by the Quartermaster Corps.

The committee also has under consideration a proposal from the aircraft industry, through the Aircraft Research and Testing Committee (A.R.T.C.) of the Aircraft Industries Association, that the Society examine the Outline for Aircraft Equipment Testing prepared by the A.R.T.C. and published by the National Aircraft Standards Committee, and determine whether the outline might be developed into an A.S.T.M. method of test. The aircraft group points out that this document, while developed for testing of equipment for the aircraft industry, has potential application to other industries by which the same or similar equipment is used, and that coordination of requirements for testing could be of great convenience to all suppliers of equipment.

While the committee has no specific recommendations to present to the Board of Directors at this time, it is expected that definite recommendations in one or more of the fields mentioned above will be made during the current year. It is the conclusion of the committee that activities in simulated service testing are most worth while and very definitely aid in supplementing the field of specific material test methods sponsored by the Society by focusing attention on the too frequently intangible service factor.

At the October meeting of the committee, the present officers were reelected.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

C. S. COLE,
Secretary.

APPENDIX VII

REPORT OF ADMINISTRATIVE COMMITTEE ON DISTRICT ACTIVITIES

Subsequent to the 1946 Annual Meeting of the Society, copies of the revised District Charter and District Manual that Administrative Committee on District Activities had formally adopted were distributed to all District officers. The Charter constitutes the authority by which the nucleus groups in the several geographic areas may henceforth function as Society representatives in their respective areas. The Manual serves these groups as a guide in good practices. To date no adverse comments have been received from any of the Districts, although word from all of the Districts in connection with these documents has not been received.

There are now eleven organized Districts: Chicago, Cleveland, Detroit, New England, New York, Northern California, Philadelphia, Pittsburgh, St. Louis, Southern California, and the Western New York-Ontario District, the latter dividing its meetings between Buffalo and Rochester, N. Y. Steps are now under way that will lead to the organization of a new District in the Washington, D. C., and Baltimore areas. Discussions and one meeting with a group of Society members in Washington have been held during the past year. Such contacts emphasize the value that may be expected to accrue to the Society and to representatives of industry and the Government in these two areas. It is expected that this new District will be organized this summer.

Study of an acceptable definition of District areas is under way. This involves the establishment of a broad policy relating to the extent to which the entire Society membership shall be affiliated with the several districts. This subject has not been clarified as yet; but it is one that is receiving current attention by the committee.

On two or three occasions the question of the District becoming affiliated with a local engineering society or group of such societies, organized to promote general engineering interest in the community, has been brought up. It has seemed wise that no general policy with respect to such affiliations be established, but rather that each such case be presented to the Administrative Committee for its consideration.

Matters to be considered by the committee in future meetings are: (*1*) The attitude to be taken toward the proposal that there be organized a national council of affiliated engineering society councils. Apparently there is something to be said on both sides of this proposal. (*2*) A strong promotional program to secure members. This could effectively be headed up in the several Districts where it has been observed that the attendance at meetings is made up of more than 50 per cent non-members, but who obviously are interested in A.S.T.M. work and subjects discussed. Cooperation with the Membership Committee of the

Board of Directors will be sought before initiating any definite program.

As was done on the occasion of the 1946 Annual Meeting, it is proposed to hold a meeting of District Chairmen, Vice-Chairmen, and Secretaries or their representatives during the 1947 Annual Meeting. Although the Annual Meetings are occasions when the membership of the Society naturally gets together to exchange views, it is felt that considerable advantage to the Society will result from a conference in which there can be an exchange of views among the leaders in the several Districts.

Respectfully submitted on behalf of the committee,

C. H. FELLOWS,
Chairman.

R. J. PAINTER,
Secretary.

REPORT OF COMMITTEE A-1*

ON

STEEL

During the past year Committee A-1 on steel has been very active in the formulation of new specifications as the result of developments in the industry and to fill in gaps in fields where specifications have been needed for some time. In 1946 a large number of widely used specifications were placed on a peacetime basis, with some of the wartime requirements deleted and many that proved to be worth while being retained. In addition, this year the committee has revised a number of existing specifications to keep abreast of the changing industrial progress and to eliminate inconsistencies which during the war years, although recognized, were permitted to exist because of the pressure of more important problems. The committee had so many problems before it this year that all could not be considered at the spring meeting held in Philadelphia in February and consequently many items were delayed for discussion and action until the Annual Meeting in Atlantic City in June. This Report of Committee A-1 accordingly was not preprinted and the recommendations on standards and tentatives covered at the Spring Meeting were distributed in mimeographed form for use at the Annual Meeting. This procedure eliminated a great deal of extensive revision which would have been necessary had the report been printed.

The committee is recommending for publication as tentative 9 new specifications[1] as covered in this report under the discussion of subcommittee activities. Revisions in 10 tentatives and 25 standards are recommended and either itemized in the Appendix to this report or discussed under the various subcommittee actions. Six tentatives are recommended for adoption as standard without change and one tentative is recommended for adoption with revisions as outlined in the Appendix. One standard is recommended for revision and reversion to tentative. These actions have been submitted to letter ballot of the committee.[2]

Subcommittee Chairmen Retiring.—At the Annual Meeting in June, E. J. Edwards, of the American Locomotive Co., announced that he was relinquishing the chairmanship of Subcommittee XI on Steel for Boilers and Pressure Vessels. Mr. Edwards agreed to carry the activities temporarily pending the naming of a successor. Chairman since 1921 and a member of the subcommittee and influential in its affairs since 1917, Mr. Edwards has reconciled many divergent viewpoints with the result that the growing number of specifications for plates for boilers and pressure vessels have come into very widespread use. Mr. Edwards' handling of the subcommittee, his integrity, and forthrightness, tempered with a spirit of understanding reflect greatly to the credit of

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] These specifications were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-A.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Committee A-1, the Society, and his company as well.

J. Hall Taylor, of the Taylor Pipe and Forge Works, relinquished the chairmanship of the Section on Forgings of Subcommittee XXII and also resigned from Subcommittee IX on Steel Tubing and Pipe. Mr. Taylor has been a member of the Society since 1923. His knowledge of the manufacture of pipe and forgings has contributed greatly to the work of the various subcommittees of which he was a member and Committee A-1 wishes to express its appreciation of his long-time activites on its behalf.

New Subcommittee.—During the past year a new Subcommittee XXVI on Common Bolting was appointed to cover a field which Committee A-1 had not approached until now. The chairman is A. D. Morris of the Bayonne Bolt Corp., and the secretary is J. J. Kelley of the Pittsburgh Screw and Bolt Co. The subcommittee consists of 26 members and has written the Specifications for Steel Machine Bolts and Nuts and Tap Bolts (A 307 – 47 T)[1] being submitted as tentative.

Subcommittee I on Steel Rails and Accessories:

At the Annual Meeting the contact groups of this subcommittee with the A.R.E.A. were reduced in number from five to two. Subgroup I, consisting of Messrs. Code, McBrian, Dudley, and Reece, will be the contact group with the A.R.E.A. Rail Committee. Subgroup II, consisting of Messrs. Kress, Crane, Trimble, and Kent, will be the contact with the A.R.E.A. Track Committee.

Changes in several specifications are being considered, but no action was taken pending A.R.E.A. approval.

Subcommittee II on Structural Steel for Bridges, Buildings and Rolling Stock:

This subcommittee has under consideration the advisability of preparing a general specification covering broad details such as terms of delivery, certain tolerances, and related matters. This document would then be referred to in a product specification, such as the one for Structural Steel for Bridges and Buildings (A 7 – 46). There is considerable material now necessary to provide complete coverage for structural steel that is not at present in Specifications A 7. Much of this information could be applicable to other structural and related specifications.

Subcommittee V on Steel Reinforcement Bars:

A new document in the form of the Tentative Specifications for Minimum Requirements for the Deformations of Deformed Steel Bars for Concrete Reinforcement (A 305 – 47 T)[1] has been prepared by this subcommittee for approval of the Society. Tentative revisions in Specifications A 15, A 16, and A 160, modifying bend test and elongation requirements applying to deformed bars shipped or ordered according to Specifications A 305 represent a complete change so far as the production of deformed bars in the industry is concerned. The subgroup which was charged with writing Specifications A 305 will continue to study the bend test requirements for bars of existing type deformations and those of special requirements covered in A 305 in order to make recommendations on the final values for bend tests.

Subcommittee VI on Steel Forgings and Billets:

Subcommittee VI has been very active during the past year reviewing the forging and billet specifications with a view toward eliminating inconsistencies and keeping abreast of industrial practice. The various revisions have been itemized in the Appendix to this report, but in addition the subcommittee recommends that the Tentative Specifications for

Carbon-Steel Seamless Drum Forgings (A 266 – 44 T) and the Tentative Methods of Magnetic Particle Testing and Inspection of Heavy Steel Forgings (A 275 – 44 T) be adopted as standard without revision.

Subcommittee IX on Steel Tubing and Pipe:

In addition to the revisions in the various specifications under the jurisdiction of this subcommittee listed in the Appendix to this report the following specifications are recommended, jointly with Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, for adoption as standard without revision:

Tentative Specifications for:

Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 44 T),

Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 44 T), and

Seamless Austenitic Chromium-Nickel Steel Tubes for Refinery Service (A 271 – 46 T).

It was also recommended that, in all the specifications under the jurisdiction of this subcommittee, all chemical analysis be designated to the second decimal place, except for the elements phosphorus and sulfur, which shall be uniformly designated to the third decimal place.

Tentative revisions (as printed in Part I-A of the 1946 Book of A.S.T.M. Standards) of the following specifications are recommended for adoption as standard:

Standard Specifications for:

Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 46),

Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 46).

Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 44),

Welded Alloy-Steel Boiler and Superheater Tubes (A 249 – 46), and

Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 46).

Subcommittee XI on Steel for Boilers and Pressure Vessels:

All the boiler plate specifications under the jurisdiction of this subcommittee are being revised as described in the Appendix to this report. In addition, the Standard Specifications for Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 46)[3] are being reverted to a tentative status with the addition of two new grades. Four new specifications have been written by the subcommittee as follows:

Tentative Specifications for:

High Tensile Strength Carbon-Manganese-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 299 – 47 T),[1]

Steel Plates for Pressure Vessels for Service at Low Temperatures (A 300 – 47 T),[1]

Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 301 – 47 T),[1] and

Manganese-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 302 – 47 T).[1]

Subcommittee XV on Bar Steels:

Subcommittee XV has gone through a very active year in extensively revising the Standard Specifications for Hot-Rolled Carbon-Steel Bars (A 107 – 46),[3] and for Cold-Finished Carbon-Steel Bars and Shafting (A 108 – 46),[3] as well as developing two new specifications. Specifications A 107 were revised by providing for both merchant and special qualities, by bringing the tolerances in line with Section 9 of the A.I.S.I. Steel Products Manual, and by eliminating the Appendix on typical tensile properties, applications, and thermal treatments. Specifications A 108 were revised in somewhat the same manner. The two new specifications are as follows:

Tentative Specifications for:

Alloy-Steel Rounds Suitable for Oil Quench-

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[3] These revised specifications were accepted by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-A

ing to End-Quench Hardenability Requirements (A 304 - 47 T),[1] and
Medium Carbon-Steel Bars to Mechanical Property Requirements (A 306 - 47 T).[1]

In addition, the subcommittee has under discussion a proposed draft for specifications for stress-relief-annealed carbon-steel bars which is expected to be submitted for approval by the Administrative Committee on Standards. The revisions of A 107 and A 108 and the three new specifications represent a new approach by the subcommittee to coverage of the field of bar steels.

Subcommittee XIX on Sheet Steel and Steel Sheets:

The specifications for sheets A 245[3] and A 246[3] have undergone a thorough overhauling by Subcommittee XIX. In A 246 the title has been changed and the scope has also been changed to line up with the latest classification for sheets. Ladle analysis has been revised to provide for the analysis of all grades of sheet and check analysis for grades A, B, and C. The tables of tolerances have been revised. The inspection clause now requires outside inspection to be done when the material is on the inspection bench. An appendix giving information on the manufacturers' standard gage has been added.

Specifications A 245 has had its title changed by the addition of the words "heavy" and "sheets." The scope clause has been changed and the revised elongation values more nearly approximate mill values. Other changes parallel those in Specifications A 246.

New Tentative Specifications for Hot-Rolled Strip of Structural Quality (A 303 - 47 T)[1] have been prepared and submitted to the Society for approval. It has been evident for some time that standardized requirements for this material would be helpful to both producer and user, and the need became pressing when changes were made in the structural steel specifications A 7 which made it undesirable to use A 7 to cover strip for structural purposes.

Subcommittee XXII on Valves, Fittings, Piping and Flanges for High Temperature and Subatmospheric Temperature:

Changes are detailed in the Appendix to this report on various specifications under the jurisdiction of Subcommittee XXII. The Tentative Specifications for Heat-Treated Carbon-Steel Bolting Material (A 261 - 44 T) is being recommended for adoption as standard without change.

Respectfully submitted on behalf of the committee,

N. L. MOCHEL,
Chairman.

HENRY WYSOR,
Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

In this Appendix recommendations are given affecting certain specifications, both standard and tentative, covering various steel products. These specifications appear in their present form in the 1946 Book of A.S.T.M. Standards, Part I-A.

Revisions in Boiler and Pressure Vessel Plate Specifications—Stress Relieving—Bend Test—Tolerances—Surface Finish and Conditioning.—In all boiler and pressure vessel plate specifications, namely:

A 30 - 46
A 129 - 46
A 201 - 46
A 202 - 46
A 203 - 46
A 204 - 46
A 212 - 46
A 225 - 46
A 285 - 46

revise the clause on stress relieving to read as follows:

> When so specified on the purchase order with the mill, test specimens representing the plates shall be stress-relieved by gradually and uniformly heating them to a temperature between 1100 and 1200 F. (or a temperature range otherwise agreed upon between the manufacturer and the fabricator), holding at temperature for at least 1 hr. per inch of thickness and cooling in still atmosphere to a temperature not exceeding 600 F.

Revise the section on bend test procedure and the table of bend test requirements to eliminate the use of the pin.

Also in all the specifications listed above revise the table on permissible overweights of plates ordered to thickness by changing the first line under column 1 (Specified Thickness, in.) from the present "$\frac{3}{16}$ to $\frac{1}{4}$, excl." to read "Up to $\frac{1}{4}$, excl." For this thickness and a width of "48 in. and under" add a permissible overweight of "7" per cent to the table. For the same thickness and a width of "132 to 144, incl." add a permissible overweight of "21" per cent to the table.

In all the boiler and pressure vessel plate specifications listed above retitle the section on finish to read "Surface Finish and Conditioning" and revise to read as follows:

> (*a*) The finished plate shall be free from injurious defects and shall have a workmanlike finish.
>
> (*b*) Plates not intended for riveted construction (see Paragraph (*e*)) may be conditioned by the manufacturer for the removal of surface imperfections or depressions on either surface by grinding, provided the ground area is well flared and the grinding does not cause the thickness to be less than the permissible minimum.
>
> (*c*) On plates intended for riveted construction the conditioning privilege described in Paragraph (*b*) does not apply unless agreed upon between the manufacturer and the purchaser.

REVISIONS OF TENTATIVES

Tentative Specifications for Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 - 46 T):

Section 2(a).—Delete the second sentence of this paragraph.

Table I.—Delete the column on grade B, acid-bessemer killed steel.

Section 9.—In Paragraphs (*b*) and (*c*) revise the fourth sentence of each to read as follows by the addition of the italicized words: "When the pipe wall thickness is $\frac{3}{4}$ in. and over, the A.S.T.M. standard 2-in. gage length test specimen shown in Fig. 1 may be used *at the option of the manufacturer.*

Section 16.—Revise to read as follows, retaining the present footnote 5:

16. (*a*) The finished pipe shall be reasonably straight and free from injurious defects. At the discretion of the inspector representing the purchaser, finished pipe shall be subject to rejection if surface defects acceptable under Paragraph (*c*) are not scattered, but appear over a large area in excess of what is considered a workmanlike finish.

(*b*) *Depth of Injurious Defects:* All defects shall be explored for depth. When the depth is in excess of $12\frac{1}{2}$ per cent of the nominal wall thickness or encroaches on the minimum wall thickness, such defects shall be considered injurious.

(*c*) *Machining or Grinding Defects Not Classified as Injurious:* Surface defects not classified as injurious shall be treated as follows:

(*1*) Pipe showing scabs, seams, laps, tears, or slivers not deeper than 5 per cent of the nominal wall thickness need not have these defects removed. If deeper than 5 per cent, such defects shall be removed by machining or grinding.

(*2*) Mechanical marks or abrasives and pits shall be acceptable without grinding or machining provided the depth does not exceed the limitations set forth in Paragraph (*b*) and if not deeper than $\frac{1}{16}$ in. If such defects are deeper than $\frac{1}{16}$ in. but not deeper than $12\frac{1}{2}$ per cent of the nominal wall thickness, they shall be removed by grinding or machining to sound metal.

(*3*) When defects have been removed by grinding or machining, the outside diameter at the point of grinding or machining may be reduced by the amount so removed. Should it be impracticable to secure a direct measurement, the wall thickness at the point of grinding, or at a defect not required to be removed, shall be determined by deducting the amount removed in grinding or the depth of the defect, from the minimum measured wall thickness at the ends of the pipe, and the remainder shall not be less than 87.5 per cent of the nominal wall thickness.

(*4*) Machining or grinding shall follow the mill's inspection of the pipe as rolled and shall be followed by supplementary visual inspection.

(*d*) *Repair by Welding:* Repair of injurious defects shall be permitted only subject to the approval of the purchaser. Welding of injurious defects in no case shall be permitted when the depth of defect exceeds $33\frac{1}{3}$ per cent of the nominal pipe wall thickness or the length of repair exceeds 25 per cent of the nominal diameter of the pipe. Defects shall be thoroughly chipped or ground out before welding. Each length of repaired pipe shall be retested hydrostatically in accordance with Section 8.

Section 17.—In the first sentence delete the phrase "where acid-bessemer steel is used, the word bessemer shall be added, that is, bessemer B."

Tentative Specifications for Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 280 – 46a T):

Section 5.—In the table of chemical composition change the chromium content from the present range of "0.40 to 0.60" to read "0.50 to 0.70" per cent.

Tentative Specifications for Carbon-Steel and Alloy-Steel Forgings for Magnetic Retaining Rings for Turbine Generators (A 288 – 46 T):

Section 6(f).—Revise the last sentence to read "the final tempering temperature shall be not less than 1100 F."

Section 7.—Revise to read as follows:

7. (*a*) The steel shall conform to the following requirements as to chemical composition:

Phosphorus, max., per cent	0.05
Sulfur, max., per cent	0.05

(*b*) The limits for elements other than phosphorus and sulfur in the composition of alloy steel shall be agreed on by the manufacturer and the purchaser.

Section 10(b).—Add a sentence to read as follows: "The crosshead speed shall not exceed $\frac{1}{8}$ in. per min. for the determination of yield strength, nor $1\frac{1}{2}$ in. per min. for determination of the tensile strength."

Tentative Specifications for Nonmagnetic Coil Retaining Rings for Turbine Generators (A 289 – 46 T):

Section 6.—Omit the note regarding minimum amounts of alloying elements.

Section 9(b).—Add a sentence to read as indicated above for Section 10(*b*) of Specifications A 288.

Table II.—In the heading for the third column change the offset value from the present "0.10" to read "0.02" per cent.

Section 10(a).—Revise to read as follows:

> (*a*) Test specimens shall be taken from prolongations or extensions of the longitudinal axis of the ring forging, and tangent to the periphery of the ring. Unless otherwise specified, prolongations or extensions shall be forged on one end of the ring for widths over 10 in., and on both ends of the ring for widths 10 in. and over. Two test specimens, spaced 180 deg. apart, as measured between radii from the longitudinal axis of the ring, shall be taken from each prolongation. When test specimens are taken from two ends, the specimens on one end shall be located 90 deg. from the specimens on the opposite end.

Section 11(a).—Revise to read as follows: "(*a*) Unless otherwise specified, two tension tests from each prolongation of each ring forging shall be made, using specimens obtained in accordance with Section 10."

Tentative Specifications for Carbon-Steel and Alloy-Steel Forgings for Pinions for Main Reduction Gears (A 291 – 46 T):

Section 1(a).—Revise the first sentence to read as follows by the addition of the italicized words: "These specifications cover normalized and tempered or quenched and tempered carbon-steel (*grade 1*) and alloy-steel (*grade 2*) forgings for pinions for main reduction gears."

Section 7.—Revise to read as follows:

> 7. (*a*) The steel for grade 1 shall conform to the following requirements as to chemical composition:

Carbon, min., per cent	0.40[a]
Manganese, min., per cent	0.55
Phosphorus, max., per cent	0.05
Sulfur, max., per cent	0.05
Silicon, min., per cent	0.15

> (*b*) The alloy steel for grade 2 shall conform to the following requirements as to chemical composition:

Phosphorus, max., per cent	0.05
Sulfur, max., per cent	0.05

> (*c*) The limits for elements other than phosphorus and sulfur in the composition of alloy steel shall be agreed on by the manufacturer and the purchaser.

Table I.—Delete the present Table I.

Table II.—Renumber as Table I and delete the reference to grade 3. For grade 2 change the size, solid diameter or thickness, in both columns from the present value of "8" to read "10" in.

Section 11(a).—Revise the table on Brinell hardness requirements to read as follows:

Grade	Size, Solid Diameter or Thickness, in.	Brinell Hardness Number
1	10 and under	170 to 223
	Over 10	159 to 212
2	All sizes	201 to 241

Tentative Specifications for Carbon-Steel and Alloy-Steel Forgings for Turbine Generator Rotors and Shafts (A 292 – 46 T):

Section 6(f).—Revise the last sentence to read "The final tempering temperature shall be not less than 1100 F."

Section 7.—Revise to read as follows:

> 7. (*a*) The steel shall conform to the following requirements as to chemical composition:

Phosphorus, max., per cent	0.05
Sulfur, max., per cent	0.05

> (*b*) The limits for elements other than phosphorus and sulfur in the composition of alloy steel shall be agreed on by the manufacturer and the purchaser.

Table I.—Delete Table I.

Table II.—Renumber as Table I and delete the tangential prolongation requirements.

Section 10.—Delete Notes 2 and 3 following Paragraph (*a*) and add a sentence to Paragraph (*b*) to read the same as indicated above for Section 10(*b*) in Specifications A 288.

Section 14.—Revise to read as follows:

> 14. (*a*) A magnetic particle test may be made by the purchaser on each finish-machined forging to demonstrate the freedom from thermal cracks

or flakes. When specified, this test shall be made at the place of manufacture.

(*b*) In making the magnetic particle test, reference shall be made to the Tentative Methods of Magnetic Particle Testing and Inspection of Heavy Forgings (A.S.T.M. Designation: A 275).

Tentative Specifications for Carbon-Steel Alloy-Steel Forgings for Turbine Rotors and Shafts (A 293 – 46 T):

Section 1.—Revise to read as follows by the addition of the italicized words:

1. These specifications cover annealed, normalized and tempered, and double normalized and tempered carbon-steel (*grade 1*) and alloy-steel (*grade 2*) forgings for turbine rotors and shafts (see Section 6).

New Section.—Add a new Section 5 to read as follows, renumbering the subsequent sections accordingly:

5. *Machinery.*—At the option of the manufacturer, forgings may either be given such preliminary heat treatment as is necessary to make them machinable, then machined, and then annealed or normalized and tempered; or they may be annealed or normalized and tempered after forging prior to any machining. In the latter case, if agreed upon by the manufacturer and the purchaser, the forgings shall be stress relieved after rough machining.

Section 5.—Renumber as Section 6. In Paragraph (*a*) revise the reference in the last sentence from the present "(see Paragraphs (*h*) and (*i*))" to read "(see Paragraph (*f*))."

Delete the present Paragraph (*f*) and reletter the present Paragraph (*i*) as new Paragraph (*f*).

Revise the last sentence of Paragraph (*g*) to read "The final tempering temperature shall be not less than 1100 F., but if required by operating temperatures this minimum tempering temperature may be increased."

Section 6.—Renumber as Section 7 and revise to read as follows:

7. (*a*) The steel for grade 1 shall conform to the following requirements as to chemical composition and vanadium may be added upon agreement or at the option of the manufacturer:

Carbon, max., per cent	0.50
Manganese, max., per cent	1.00
Phosphorus, max., per cent	0.05
Sulfur, max., per cent	0.05
Silicon, min., per cent	0.15

(*b*) The alloy steel for grades 2 to 6, incl., shall conform to the following requirements as to chemical composition:

Phosphorus, max., per cent	0.05
Sulfur, max., per cent	0.05

(*c*) The limits for elements other than phosphorus and sulfur in the composition of alloy steel (grades 2 to 6, incl.) shall be agreed upon by the manufacturer and the purchaser. When forgings for rotors are to be used at high operating temperatures, and when specified on the drawings or order, the steel used shall contain 0.40 to 0.60 per cent molybdenum.

Table I.—Delete the present Table I.

Table II.—Renumber as Table I.

Section 9.—Renumber as Section 10 and add a sentence to Paragraph (*b*) to read as indicated above for Section 10(*b*) in Specifications A 288.

Section 13.—Renumber as Section 14 and revise the first sentence of Paragraph (*a*) to read "Unless otherwise specified by the purchaser, each forging shall be subjected to a heat indication test, after machining the forging to dimensions specified in accordance with Section 17, to determine its stability or freedom from tendency to distort during high-temperature operating conditions."

Section 14.—Renumber as Section 15 and revise to read as indicated above for Section 14 in Specifications A 292.

Tentative Specifications for Carbon-Steel and Alloy-Steel Forgings for Turbine Bucket Wheels (A 294 – 46 T):

Section 1.—Revise to read as follows by the addition of the italicized words:

1. These specifications cover annealed or annealed, quenched, and tempered carbon-steel (*grades A1 and A2*) and alloy steel (*grades B1, B2, B3 and B4*) forgings for turbine bucket wheels.

Section 6(f).—Revise the last sentence to read "The final tempering temperature shall be not less than 1100 F."

Section 7.—Revise to read as follows:

7. (*a*) The steel for grades A1 and A2 shall conform to the following requirements as to chemical composition and vanadium may be added upon agreement or at the option of the manufacturer:

Carbon, max., per cent	0.50
Manganese, max., per cent	1.00
Phosphorus, max., per cent	0.05
Sulfur, max., per cent	0.05
Silicon, min., per cent	0.15

(*b*) The alloy steel for grades B1 to B4, incl., shall conform to the following requirements as to chemical composition:

Phosphorus, max., per cent	0.05
Sulfur, max., per cent	0.05

(*c*) The limits for elements other than phosphorus and sulfur in the composition of alloy steel (grades B1 to B4, incl.) shall be agreed upon by the manufacturer and the purchaser. When forgings for rotors are to be used at high operating temperatures, and when specified on the drawings or order, the steel used shall contain 0.40 to 0.60 per cent molybdenum.

Table I.—Delete Table 1.

Table II.—Renumber as Table I and revise to read as shown in the accompanying Table I.

TABLE I.—TENSILE PROPERTIES.

Grade	Tensile Strength, min., psi.	Yield Strength (0.01 per cent offset), min., psi.	Elongation in 2 in., min., per cent	Reduction of Area, min., per cent
A1	70 000	40 000	20	50
A2	80 000	50 000	18	45
B1	100 000	70 000	20	50
B2	110 000	85 000	19	48
B3	120 000	105 000	18	45
B4	150 000	125 000	15	40

Section 10(b).—Add a sentence to read as indicated above for Section 10(*b*) in Specifications A 288.

Section 11.—Revise the table of Brinell hardness requirements to read as follows:

Grade	Brinell Hardness Number
A1	131 to 183
A2	149 to 201
B1	192 to 241
B2	207 to 255
B3	229 to 277
B4	293 to 341

TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Billet-Steel Bars for Concrete Reinforcement (A 15 - 39):

Section 5.—In the table of chemical composition change the maximum phosphorus content of acid-bessemer steel from the present value of "0.10" to "0.11" per cent.

Table I.—Add a footnote letter *b* after "Deformed Bars" and add the following under the table as footnote *b*:

Where deformations conforming to the Tentative Specifications for Minimum Requirements for the Deformations of Deformed Steel Bars for Concrete Reinforcement (A.S.T.M. Designation: A 305)[1] are specified in the order or supplied by the producer, certain changes in elongation shall be accepted as follows:

	Structural Grade	Intermediate Grade	Hard Grade
Elongation in 8 in., min., per cent	$\frac{1\,200\,000}{\text{tens. str.}}$ but not less than 16 per cent	$\frac{1\,100\,000}{\text{tens. str.}}$ but not less than 12 per cent	$\frac{975\,000}{\text{tens. str.}}$ ....

Table II.—Add a footnote letter *a* after "Deformed Bars" and add the following under the table as footnote *a*:

Where deformations conforming to the Tentative Specifications for Minimum Requirements for the Deformations of Deformed Steel Bars for Concrete Reinforcement (A.S.T.M. Designation: A 305)[1] are specified in the order or supplied by the producer, certain changes in bend test requirements shall be accepted as follows:

Thickness or Diameter of Bar	Structural Grade	Intermediate Grade	Hard Grade
Under ¾ in.	180 deg. $d = 2t$	180 deg. $d = 6t$	90 deg. $d = 6t$
¾ in. or over	180 deg. $d = 4t$	90 deg. $d = 6t$	90 deg. $d = 6t$

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[1] 1947 Supplement to Book of A.S.T.M. Standards, Part I-A.

Standard Specifications for Rail-Steel Bars for Concrete Reinforcement (A 16 – 35):

Section 4(a).—Add a footnote letter *b* after the word "Deformed" in the heading for the last column in the table of tensile properties and add the following under the table as footnote *b*:

> Where deformations conforming to the Tentative Specifications for Minimum Requirements for the Deformations of Deformed Steel Bars for Concrete Reinforcement (A.S.T.M. Designation: A 305)[1] are specified in the order or supplied by the producer, certain changes in elongation shall be accepted as follows:

	Deformed Bars
Elongation in 8 in., min., per cent...	$\frac{975\,000}{\text{tens. str.}}$

Section 5(a).—Add a footnote letter *a* after the word "Deformed" in the heading for the last column in the table of bend test requirements and add the following under the table as footnote *a*:

> Where deformations conforming to the Tentative Specifications for Minimum Requirements for the Deformations of Deformed Steel Bars for Concrete Reinforcement (A.S.T.M. Designation: A 305)[1] are specified in the order or supplied by the producer, certain changes in bend test requirements shall be accepted as follows:

Thickness or Diameter of Bar	Deformed Bars
Under $\frac{3}{4}$ in.	90 deg. d = 6t
$\frac{3}{4}$ in. or over	90 deg. d = 6t

Standard Specifications for Boiler and Firebox Steel for Locomotives (A 30 – 46):

Section 13.—Revise Section 13 on marking to read as follows:

> 13. (*a*) The name or brand of the manufacturer, or the manufacturer's test identification, class and minimum of the range of the tensile strength specified in Section 6 for the grade ordered and, in addition, the railroad customer's special identification serial numbers shall be legibly stamped on each finished plate in two places not less than 12 in. from the edges and on each butt strap near the center line not less than 12 in. from each end. Plates, the maximum lengthwise and crosswise dimensions of which do not exceed 48 in., shall have the marking stamped in one place approximately midway between the center and an edge. The manufacturer's test identification number shall be legibly stamped on each test specimen.
>
> (*b*) Except when otherwise arranged with the manufacturer (see Paragraph (*e*)) plate shall be match-marked as defined in Paragraph (*c*), so that test specimens representing them may be identified. When more than one plate is sheared from a single slab or ingot, each plate shall be match-marked so that they all may be identified with the test specimen representing them.
>
> (*c*) Each match-mark shall consist of two overlapping circles each not less than $1\frac{1}{2}$ in. in diameter, placed upon shear lines, and made by separate impressions of a single circle steel die.
>
> (*d*) Match-marked coupons, except as provided for in Paragraph (*e*), shall match with the plates represented and only those which match-mark properly shall be accepted.
>
> (*e*) When arranged with the manufacturer (see Paragraph (*b*)), for plates rolled on mills with rotary side and automatic end shears, and for plates necessitating flame cutting because of thickness or circular plates, making match-marking impracticable, the manufacturer will furnish an affidavit, in lieu of the match-marking requirement, stating that the test coupons are representative of the plates and patterns cut from the material as indicated. If the plan of furnishing an affidavit is not satisfactory, the manufacturer and the purchaser may mutually agree on some other method of test identification.

Standard Specifications for Axle-Steel Bars for Concrete Reinforcement (A 160 – 39):

Tables I and II.—Revise as indicated above for Tables I and II in Specifications A 15.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that these specifications, revised as follows, be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Tubing for the Dairy and Food Industry (A 270 – 44 T):

Title.—Change the title to read

"Standard Specifications for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing."

Section 1.—Revise Paragraphs (*a*) and (*b*) to read as follows by the addition of the italicized words and the deletion of those in parentheses:

> (*a*) These specifications cover a grade of seamless and welded austenitic stainless steel *sanitary* tubing intended for use in the dairy and food industry, in sizes up to and including 4 in. in outside diameter.
>
> (*b*) These tubes *are* (may be) furnished with special surface finishes and *therefore* the finish shall be specified in the order.

Table I.—Delete the letters "TP" before the identification symbol "304".

Section 6(*b*).—Revise to read as follows by the deletion of the words in parentheses: "(*b*) (If the purchaser specifies a polished finish surface) the tubes shall be hydrostatically tested prior to the polishing operation."

Section 8.—Delete Paragraph (*c*) and revise Paragraph (*b*) to read as follows by the addition of the italicized words and the deletion of the words in parentheses: "(*b*) (For tubes furnished "not polished,") the wall thickness at any point shall not be less than *87.5* (90) per cent of the specified average wall thickness."

Section 9.—Revise Paragraph (*a*) to read as follows by deleting the last two sentences of the present Paragraph (*a*): "(*a*) Finished tubes shall be reasonably straight and have smooth ends free from burrs. They shall be free from injurious defects and shall have a workmanlike finish."

Delete the present Paragraph (*b*) and substitute the following: "(*b*) For tubes polished on the inside surface only, the outside surface may have minor defects removed by grinding, provided the wall thicknesses are not decreased to less than that prescribed in Section 8(*b*)."

In Paragraph (*c*) delete Finish P and the note and add two new sentences to read as follows: "All of the above finishes can be produced internally or externally on the tube surface and combinations of these four finishes can be obtained. For tubes polished on the inside surface only, the outside surface shall be finished with regular pickle finish."

Table II.—Revise Table II to read as shown in the accompanying Table II.

TABLE II.—PERMISSIBLE VARIATIONS IN DIMENSIONS.

Size, Outside Diameter, in.	Permissible Variations in Outside Diameter, in.		Permissible Variations in Cut Length, in.	
	Over	Under	Over	Under
1	0.002	0.008	⅛	0
1½	0.002	0.008	⅛	0
2	0.002	0.011	⅛	0
2½	0.002	0.011	⅛	0
3	0.003	0.012	⅛	0
4	0.003	0.015	⅛	0

Revisions of Standards, Immediate Adoption

Standard Specifications for Carbon-Steel Axles for Cars and Tenders (A 21 – 36):

Section 3.—Revise the table of chemical composition as follows:

	Present Values	Revised Values
Carbon, per cent	0.40 to 0.55	0.40 to 0.55
Manganese, per cent	0.50 to 0.90	0.60 to 0.90
Phosphorus, max., per cent	0.05	0.045
Sulfur, max., per cent	0.06	0.05

Section 8(*b*).—Revise to read as follows: "(*b*) When less than 15 tons of any one design is ordered, the manufacturer shall have the option of furnishing the purchaser a certified copy of a previous test of the heat."

Section 11.—Letter the present as Paragraph (*a*) and add a new Paragraph (*b*) to read as follows: "(*b*) Any straightening of unmachined axles, if required, shall be done at a temperature of not less than 950 F. and in such a manner as to leave the surface free from objectionable scars."

Standard Specifications for Boiler and Firebox Steel for Locomotives (A 30 – 46):

Section 6(d).—Add the following to this sentence: "and the elongation shall conform to the minimum requirement specified for the 8 in. gage length."

Standard Specifications for Welded and Seamless Steel Pipe (A 53 – 46):

Section 1(a).—Revise to read as follows:

1. (*a*) These specifications cover black and hot-dipped-galvanized welded and seamless steel pipe for the following purposes:

(*1*) Pipe ordered under these specifications is nominal (average) wall and is intended for coiling, bending, flanging, and other special purposes, and is suitable for welding.

(*2*) When seamless or electric-resistance-welded pipe is ordered for close coiling, cold bending, and for forge welding, grade A should be specified, as grade B is not intended for these purposes.

(*3*) Butt-welded pipe is not intended for flanging.

(*4*) The purpose for which the pipe is intended should be stated in the order.

Footnote 2.—Delete footnote 2.

Section 2(a).—Revise to read as follows:

(*a*) The steel for both welded and seamless pipe shall be made by one or more of the following processes: open-hearth, electric-furnace, or acid-bessemer, except that steel for grade B bessemer pipe shall be killed steel made by the deoxidized acid-bessemer process (Note), and electric-resistance-welded pipe ⅛ and ¼ in. in diameter shall be made from open-hearth steel. The steel for furnace-welded pipe shall be of soft weldable quality.

NOTE.—Deoxidized bessemer steel for seamless pipe is an acid-bessemer steel which has had a considerable portion of dissolved oxygen removed as a gas by treatment in the vessel and further treated in the ladle to develop a silicon content of 0.10 to 0.30 per cent and with sufficient aluminum, or its equivalent, to insure practically no evolution of gases during solidification.

Section 8.—Revise to read as follows:

8. Each length of pipe shall be tested at the mill to the hydrostatic pressures prescribed in Tables II, III, IV or V (accompanying Tables III, IV, V, and VI). The hydrostatic test may be applied at the discretion of the manfacturer on pipe with plain ends, with threads only, or with threads and couplings. Welded pipe 2 in. and larger shall be jarred near one end while under test pressure.

TABLE III.—DIMENSIONS, NOMINAL WEIGHTS, (PLAIN ENDS AND THREADS^{ac} AND COUPLINGS^{bc}) AND TEST PRESSURES FOR "STANDARD WEIGHT" PIPE (SCHEDULE 40).

NOTE.—Pipe furnished in accordance with this table will be "Standard Pipe," which is defined in Section 18 of the AISI Steel Products Manual as welded or seamless pipe made in three classes of wall thickness: (*1*) *standard weight*, in nominal sizes ⅛ to 6 in., inclusive, (*2*) *extra strong*, in nominal sizes ⅛ to 12 in., inclusive, and (*3*) *double extra strong*, in nominal sizes ½ to 8 in., inclusive.

Size, in.	Outside Diameter, in.	Wall Thickness, in.	Weight per Foot, lb.		Test Pressure, psi.		
			Plain Ends	Threads and Couplings	Butt-Welded	Lap-Welded^e or Grade A^{fg}	Grade B^{fg}
⅛	0.405	0.068	0.24	0.24	700	700	700
¼	0.540	0.088	0.42	0.42	700	700	700
⅜	0.675	0.091	0.57	0.57	700	700	700
½	0.840	0.109	0.85	0.85	700	700	700
¾	1.050	0.113	1.13	1.13	700	700	700
1	1.315	0.133	1.68	1.68	700	700	700
1¼	1.660	0.140	2.27	2.28	800	1000	1100
1½	1.900	0.145	2.72	2.73	800	1000	1100
2	2.375	0.154	3.65	3.68	800	1000	1100
2½	2.875	0.203	5.79	5.82	800	1000	1100
3	3.500	0.216	7.58	7.62	800	1000	1100
3½	4.000	0.226	9.11	9.20	1200	1200	1300
4	4.500	0.237	10.79	10.89	1200	1200	1300
5	5.563	0.258	14.62	14.81	[d]	1200	1300
6	6.625	0.280	18.97	19.18	[d]	1200	1300

[a] The taper of threads on pipe is ¾ in. per foot on the diameter for all sizes. See Section 15(*b*), Table IX (accompanying Table VII), and Fig 1.

[b] See Section 15(*c*), Table IX (accompanying Table VII), and Fig. 1.

[c] For illustration of joint and detailed threading data, see Table IX (accompanying Table VII) and Fig. 1. Threading data shown therein are identical with corresponding portions of the American Standard for Pipe Threads (ASA No.: B2.1-1945).

[d] Butt-welded pipe is not made in sizes larger than 4 in.

[e] Lap-welded pipe is not manufactured in sizes under 2 in.

[f] Seamless pipe in some of the smaller sizes may be cold drawn.

[g] Grades A and B refer to seamless or electric welded pipe.

Tables II and III.—Delete the present Tables II and III.

New Tables.—Add new Tables II, III, IV, and V as shown in the accompanying Tables III, IV, V, and VI.

Section 15.—Revise Paragraphs (*b*) and (*c*) to read as follows:

(*b*) *Threads.*—All threads shall be in accordance with the gaging practice of the American Standard for Pipe Threads.[5] The variation from the standard, when tested with the standard working gage, shall not exceed 1½ turns either way

(*c*) *Couplings.*—Each length of threaded pipe shall be provided with one coupling, the threads of which shall be in accordance with the gaging practice of the American Standard for Pipe Threads.[5] The coupling shall be applied handling-tight. If so specified, coupilngs will be applied power-tight. Couplings may be wrought iron or steel. Taper tapped couplings shall be furnished on all weights of threaded pipe 2½ in. and larger.[6]

TABLE IV.—DIMENSIONS, NOMINAL WEIGHTS (PLAIN ENDS AND THREADS[a,c,d] AND COUPLINGS[b,c,d]), AND TEST PRESSURES FOR PIPE 8 IN. AND LARGER (SCHEDULES 30 AND 40).

Size, in.[e]	Outside Diameter, in.	Wall Thickness, in.[f,g]	Weight per Foot, lb.[g]		Test Pressure, psi.	
			Plain Ends	Threads and Couplings	Lap Welded or Grade A[k]	Grade B[k]
8	8.625	0.277	24.70	25.55	1200	1300
8[h]	8.625	0.322	28.55	29.35	1300	1600
10[i]	10.750	0.279	31.20	32.75	1000	1200
10	10.750	0.307	34.24	35.75	1000	1200
10[h]	10.750	0.365	40.48	41.85	1200	1400
12	12.750	0.330	43.77	45.45	1000	1200
12[j]	12.750	0.375	49.56	51.15	1100	1200

[a] The taper of threads on pipe is ¾ in per foot on the diameter for all sizes. See Section 15(*b*), Table X (accompanying Table VIII), and Fig. 2

[b] See Section 15(*c*), Table X (accompanying Table VIII), and Fig. 2

[c] For illustration of joint and detailed threading data, see Table X (accompanying Table VIII) and Fig. 2. Threading data shown therein are identical with corresponding portions of the American Standard for Pipe Threads (ASA No.: B2.1 1945)

[d] If threads and couplings are required, the pipe is furnished with recessed couplings.

[e] Sizes larger than those shown in the table are measured by their outside diameter. These larger sizes will be furnished with plain ends, unless otherwise specified. The weights will correspond to the manufacturers' published standards although it is possible to calculate the theoretical weights for any given size and wall thickness on the basis of 1 cu. in. of steel weighing 0.2833 lb

[f] Wall thicknesses other than shown, such as covered by Schedules 10, 20, 60, etc., are a matter of agreement between the purchaser and the manufacturer

[g] As more than one weight is listed under the same size the order must definitely specify both the weight and wall thickness required.

[h] Schedule 40 pipe.

[i] Ten inch pipe with a 0.279-in. wall is not covered by a schedule number.

[j] Owing to a departure from the wall thickness for 12-in size, Schedule 40 of the American Standard for Wrought-Iron and Wrought-Steel Pipe (ASA No.: B36.10-1939), the wall thickness 0.375 may be substituted for 0.406 where agreeable to the purchaser and suitable for the service conditions.

[k] Grades A and B refer to seamless or electric welded pipe.

TABLE V.—DIMENSIONS, NOMINAL WEIGHTS (PLAIN ENDS AND THREADS[a,c,d] AND COUPLINGS[b,c,d]), AND TEST PRESSURES FOR "EXTRA STRONG" PIPE (SCHEDULE 80).

NOTE.—Pipe furnished in accordance with this table will be "Standard Pipe," which is defined in Section 18 of the AISA Steel Products Manual as welded or seamless pipe made in three classes of wall thickness: (*1*) *standard weight*, in nominal sizes ⅛ to 6 in., inclusive, (*2*) *extra strong*, in nominal sizes ⅛ to 12 in., inclusive, and (*3*) *double extra strong*, in nominal sizes ½ to 8 in., inclusive.

Size, in.	Outside Diameter, in.	Wall Thickness, in.	Weight per Foot, lb. Plain Ends	Test Pressure, psi.		
				Butt-Welded	Lap-Welded[f] or Grade A[g,j]	Grade B[g,j]
⅛	0.405	0.095	0.31	850	850	850
¼	0.540	0.119	0.54	850	850	850
⅜	0.675	0.126	0.74	850	850	850
½	0.840	0.147	1.09	850	850	850
¾	1.050	0.154	1.47	850	850	850
1	1.315	0.179	2.17	850	850	850
1¼	1.660	0.191	3.00	1100	1500	1600
1½	1.900	0.200	3.63	1100	1500	1600
2	2.375	0.218	5.02	1100	1500	1600
2½	2.875	0.276	7.66	1100	1500	1600
3	3.500	0.300	10.25	1100	1500	1600
3½	4.000	0.318	12.51	1700	1700	1800
4	4.500	0.337	14.98	1700	1700	1800
5	5.563	0.375	20.78	[e]	1700	1800
6	6.625	0.432	28.57	[e]	1700	1800
8	8.625	0.500	43.39	[e]	1700	2400
10[h]	10.750	0.500	54.74	[e]	1600	1900
12[i]	12.750	0.500	65.42	[e]	1600	1900

[a] The taper of threads on pipe is ¾ in. per foot on the diameter for all sizes. See Section 15(*b*), Table X (accompanying Table VIII), and Fig. 2.

[b] See Section 15(*c*), Table X (accompanying Table VIII), and Fig. 2.

[c] For illustration of joint and detail threading data, see Table X (accompanying Table VIII), and Fig. 2. Threading data shown therein are identical with corresponding portions of the American Standard for Pipe Threads (ASA No. B2.1-1945).

[d] If threads and couplings are required, the pipe is furnished with recessed couplings.

[e] Butt-welded pipe is not made in sizes larger than 4 in.

[f] Lap-welded pipe is not manufactured in sizes under 2 in.

[g] Seamless pipe in some of the smaller sizes may be cold drawn.

[h] Schedule 60 pipe.

[i] Owing to a departure from the wall thickness for the 12-in. size, Schedule 60 of the American Standard for Wrought-Iron and Wrought Steel Pipe (ASA No. B36.10-1939), the wall thickness of 0.500 may be substituted for 0.562 where agreeable to the purchaser and suitable for the service conditions.

[j] Grades A and B refer to seamless or electric welded pipe.

Footnote 5.—Revise to read as follows:

A complete description of the American Standard Pipe Threads applicable to pipe, valves and fittings is contained in the American Standard for Pipe Threads (ASA No.: B2.1-1945). "Screw Thread Standards for Federal Services," National Bureau of Standards *Handbook H-28* (1944), also contains the same pertinent data, both sources being identical.

TABLE VI.—DIMENSIONS, NOMINAL WEIGHTS (PLAIN ENDS AND THREADS[a, d] AND COUPLINGS[b c d]), AND TEST PRESSURES FOR "DOUBLE EXTRA STRONG[h]" PIPE.

NOTE.—Pipe furnished in accordance with this table will be "Standard Pipe," which is defined in Section 18 of the AISI Steel Products Manual as welded or seamless pipe made in three classes of wall thickness: (*1*) *standard weight*, in nominal sizes ⅛ to 6 in., inclusive, (*2*) *extra strong*, in nominal sizes ⅛ to 12 in., inclusive, and (*3*) *double extra strong*, in nominal sizes ½ to 8 in., inclusive.

Size, in.	Outside Diameter, in.	Wall Thickness, in.	Weight per Foot, Plain Ends, lb.	Test Pressure, psi.		
				Butt-Welded	Lap-Welded[f] or Grade A[g i]	Grade B[g i]
½	0.840	0.294	1.71	1000	1000	1000
¾	1.050	0.308	2.44	1000	1000	1000
1	1.315	0.358	3 66	1000	1000	1000
¼	1.660	0.382	5.21	1200	1800	1900
1½	1.900	0.400	6.41	1200	1800	1900
2	2.375	0.436	9.03	1200	1800	1900
2½	2.875	0.552	13.70	1200	1800	1900
3	3.500	0.600	18.58	[e]	1800	1900
4	4.500	0.674	27.54	[e]	2000	2100
5	5.563	0.750	38.55	[e]	2000	2100
6	6.625	0.864	53.16	[e]	2000	2100
8	8.625	0.875	72.42	[e]	2800	2800

[a] The taper of threads on pipe is ¾ in. per foot on the diameter for all sizes. See Section 15(*b*), Table X (accompanying Table VIII), and Fig. 2.

[b] See Section 15(*c*), Table X (accompanying Table VIII), and Fig. 2.

[c] For illustration of joint and detail threading data, see Table X (accompanying Table VIII), and Fig. 2. Threading data shown therein are identical with corresponding portions of the American Standard for Pipe Threads (ASA No.: B2 1-1945).

[d] If threads and couplings are required, the pipe is furnished with recessed couplings.

[e] Butt-welded pipe is not made in sizes larger than 4 in.

[f] Lap-welded pipe is not manufactured in sizes under 2 in.

[g] Seamless pipe in some of the smaller sizes may be cold drawn.

[h] The American Standard for Wrought-Iron and Wrought-Steel Pipe (ASA No.: B36.10-1939) has assigned no schedule number to "Double Extra Strong" pipe.

[i] Grades A and B refer to seamless or electric welded pipe.

New Appendix.—Add a new Appendix IV to include new Tables IX and X (accompanying Tables VII and VIII) and the accompanying Figs. 1 and 2.

Standard Specifications for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 46):

Tables I and II.—Delete the present Tables I and II.

New Tables.—Add new Tables I, II, III, and IV as shown in the accompanying Tables III, IV, V, and VI.

Section 4.—Revise to read as indicated above for Section 8 in Specifications A 53, referring to Tables I, II, III, and IV (accompanying Tables III, IV, V, and VI).

Section 13.—Revise Paragraphs (*b*) and (*c*) to read as indicated above for Section 15 in Specifications A 53.

Footnote 5.—Revise to read as indicated above for footnote 5 in Specifications A 53.

New Appendix.—Add a new Appendix II as indicated above for the new Appendix in Specifications A 53. The accompanying Tables VII and VIII will be numbered Tables VI and VII.

Standard Specifications for Carbon-Silicon Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 46):

Title.—Change the title to read as follows by the addition of the italicized word and the deletion of the word in parentheses: "Standard Specifications for Carbon-Silicon Steel Plates of *Intermediate* (Ordinary) Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels."

Section 1.—Revise the first and second sentences to read as follows: "These specifications cover carbon-silicon steel plates, in two intermediate tensile strength ranges designated grades A and B, intended particularly for fusion welding; for use in locomotive boiler shells, boilers for stationary service and other pressure vessels. The maximum thickness of flange quality plates to be specified under these specifications shall be 2 in.; of firebox quality plates of grade A shall be 12 in.; and of grade B shall be 8 in.

Section 3(a).—Transpose the second and third sentences.

Section 4.—In the table of chemical composition revise the carbon content requirements to read as follows:

	Grade A	Grade B
Carbon, max. per cent:		
For plates 1 in. and under in thickness	0.20	0.24
For plates over 1 to 2 in., incl., in thickness	0.24	0.27
For plates over 2 to 4 in., incl., in thickness	0.27	0.30
For plates over 4 to 8 in., incl., in thickness	0.31	0.35
For plates over 8 to 12 in., incl., in thickness	0.35	....

Section 7(g).—Revise to read as indicated above for Section 6(*d*) in Specifications A 30.

TABLE VII.—BASIC THREADING DATA FOR PIPE PRESCRIBED IN TABLE II (ACCOMPANYING TABLE III).

NOTE 1.—All dimensions in this table are nominal and subject to mill tolerances.
NOTE 2.—The taper of threads is ¾ in. per foot on the diameter.

Pipe		Threads					Coupling		
Nominal Size, in.	Outside Diameter, in. D	Number per Inch	End of Pipe to Hand Tight Plane, in. L_1	Effective Length, in. L_2	Total Length, in. L_4	Pitch Diameter at Hand Tight Plane, in. E_1	Outside Diameter, in. W	Length, in. N_L	Hand Tight Stand-Off (Number of Threads) A
⅛	0.405	27	0.180	0.2638	0.3924	0.37476	0.563	13/16	4
¼	0.540	18	0.200	0.4018	0.5946	0.48989	0.719	1 3/16	5½
⅜	0.675	18	0.240	0.4078	0.6006	0.62701	0.875	1 3/16	5
½	0.840	14	0.320	0.5337	0.7815	0.77843	1.063	1 9/16	5
¾	1.050	14	0.339	0.5457	0.7935	0.98887	1.313	1⅝	5
1	1.315	11½	0.400	0.6828	0.9845	1.23863	1.576	2	5
1¼	1.660	11½	0.420	0.7068	1.0085	1.58338	1.900	2 1/16	5
1½	1.900	11½	0.420	0.7235	1.0252	1.82234	2.200	2 1/16	5½
2	2.375	11½	0.436	0.7565	1.0582	2.29627	2.750	2⅛	5½
2½	2.875	8	0.682	1.1375	1.5712	2.76216	3.250	3⅛	5½
3	3.500	8	0.766	1.2000	1.6337	3.38850	4.000	3¼	5½
3½	4.000	8	0.821	1.2500	1.6837	3.88881	4.625	3⅜	5½
4	4.500	8	0.844	1.3000	1.7337	4.38713	5.000	3½	5
5	5.563	8	0.937	1.4063	1.8400	5.44929	6.296	3¾	5
6	6.625	8	0.958	1.5125	1.9462	6.50597	7.390	4	6

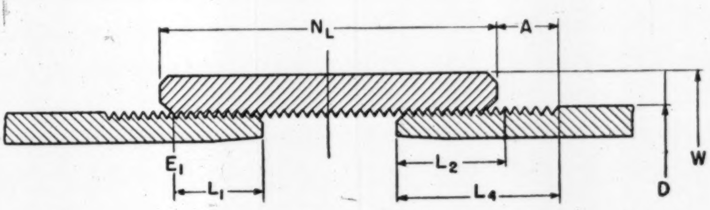

FIG. 1.—Hand Tight Assembly Dimensions for Use with Accompanying Table VII.

Standard Specifications for Chromium-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels (A 202 – 47):

Section 6(e).—Revise to read as indicated above for Section 6(*d*) in Specifications A 30.

Standard Specifications for Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 – 46):

Section 3(a).—Revise as indicated above for Section 3(*a*) in Specifications A 201.

Section 7(g).—Revise as indicated above for Section 6(*d*) in Specifications A 30.

Standard Specifications for High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (Plates 4½ in. and Under in Thickness) (A 212 – 46):

Title.—Revise the title to read as

TABLE VIII.—BASIC THREADING DATA FOR PIPE PRESCRIBED IN TABLES III, IV, AND V (ACCOMPANYING TABLES IV, V, AND VI).

NOTE.—The taper of threads is ¾ in. per foot on the diameter.

Pipe		Threads					Coupling		
Nominal Size, in.	Outside Diameter, in. *D*	Number per Inch	End of Pipe to Hand Tight Plane, in. *L*₁	Effective Length, in. *L*₂	Total Length, in. *L*₄	Pitch Diameter at Hand Tight Plane, in. *E*₁	Outside Diameter, in. *W*	Length, in. *N L*	Hand Tight Stand-off (Number of Threads) *A*
⅛	0.405	27	0.180	0.2638	0.3924	0.37476	0.563	1 1/16	3
¼	0.540	18	0.200	0.4018	0.5946	0.48989	0.719	1⅝	3
⅜	0.675	18	0.240	0.4078	0.6006	0.62701	0.875	1⅝	3
½	0.840	14	0.320	0.5337	0.7815	0.77843	1.063	2⅛	3
¾	1.050	14	0.339	0.5457	0.7935	0.98887	1.313	2⅛	3
1	1.315	11½	0.400	0.6828	0.9845	1.23863	1.576	2⅝	3
1¼	1.660	11½	0.420	0.7068	1.0085	1.58338	2.054	2¾	3
1½	1.900	11½	0.420	0.7235	1.0252	1.82231	2.200	2¾	3
2	2.375	11½	0.436	0.7565	1.0582	2.29627	2.875	3¼	3
2½	2.875	8	0.682	1.1375	1.5712	2.76216	3.375	4⅛	2
3	3.500	8	0.766	1.2000	1.6337	3.38850	4.000	4¼	2
3½	4.000	8	0.821	1.2500	1.6837	3.88881	4.625	4⅜	2
4	4.500	8	0.844	1.3000	1.7337	4.38713	5.200	4½	2
5	5.563	8	0.937	1.4063	1.8400	5.44929	6.296	4⅝	2
6	6.625	8	0.958	1.5125	1.9462	6.50597	7.390	4⅞	2
8	8.625	8	1.063	1.7125	2.1462	8.50003	9.625	5¼	2
10	10.750	8	1.210	1.9250	2.3587	10.62094	11.750	5¾	2
12	12.750	8	1.360	2.1250	2.5587	12.61781	14.000	6⅛	2
14[a]	14.000	8	1.562	2.2500	2.6837	13.87263	15.000	6⅜	2
16[a]	16.000	8	1.812	2.4500	2.8837	15.87575	17.000	6¾	2
18[a]	18.000	8	2.000	2.6500	3.0837	17.87500	19.000	7⅛	2
20[a]	20.000	8	2.125	2.8500	3.2837	19.87031	21.000	7⅝	2

[a] Nominal size is outside diameter.

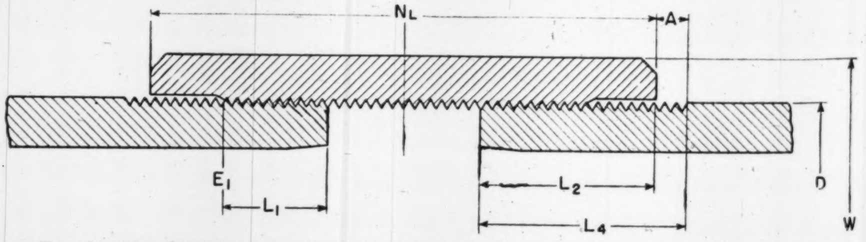

FIG. 2.—Hand Tight Assembly Dimensions for Use with Accompanying Table VIII.

follows: "Standard Specifications for High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (Plates 6 in. and Under in Thickness)."

Section 1.—Revise the first and second sentences to read as follows: "These specifications cover carbon-silicon steel plates in two high tensile strength ranges, designated as grades A and B, of flange and firebox qualities; for use in locomotive boiler shells, boilers for stationary service, and other pressure vessels. The maximum thickness of flange quality plates to be specified under these specifications shall be 2 in.; and for firebox quality, 6 in."

Section 4.—In the table of chemical composition revise the carbon content values to read as follows:

	Grade A	Grade B
Carbon, max., per cent:		
For plates 1 in. and under in thickness	0.28	0.31
For plates over 1 to 2 in., incl., in thickness	0.31	0.33
For plates over 2 to 6 in., incl., in thickness	0.33	0.35

Section 7(f).—Revise as indicated above for Section 6(*d*) in Specifications A 30.

Section 8.—In the table of bend test requirements add the following:

Thickness of Material	Ratio of Pin Diameter to Thickness of Specimen Grade A	Ratio of Pin Diameter to Thickness of Specimen Grade B
Over $4\frac{1}{2}$ to 6 in., incl.	3	$3\frac{1}{2}$

Table II.—Revise the last line of the first column from the present range of "4 to $4\frac{1}{4}$, incl." to read "4 to 6, excl." and add the following to the table:

Specified Thickness, in.	Variations over Specified Thickness for Widths Given, in.					
	Up to 36, excl.	36 to 60, excl.	60 to 84, excl.	84 to 120, excl.	120 to 132, excl.	132 and over
6	7/64	1/8	5/32	5/32	11/64	..

Standard Specifications for Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 – 46):

Section 3(a).—Revise as indicated above for Section 3(*a*) in Specifications A 201.

Section 7(f).—Revise as indicated above for Section 6(*d*) in Specifications A 30.

Standard Specifications for Hard-Drawn Steel Spring Wire (A 227 – 41):

Table I.—Revise Table I to read as shown in the accompanying Table IX.

TABLE IX.—TENSILE REQUIREMENTS.

Diameter, in.	Tensile Strength, psi.		Diameter, in.	Tensile Strength, psi.	
	Min.	Max.		Min.	Max.
0.028	271 000	311 000	0.148	203 000	234 000
0.032	265 000	305 000	0.162	200 000	230 000
0.035	261 000	301 000	0.177	195 000	225 000
0.041	255 000	293 000	0.192	192 000	221 000
0.047	248 000	286 000	0.207	190 000	218 000
0.054	243 000	279 000	0.225	186 000	214 000
0.0625	237 000	272 000	0.250	182 000	210 000
0.072	232 000	266 000	0.312	174 000	200 000
0.080	227 000	261 000	0.375	167 000	193 000
0.093	220 000	253 000	0.437	165 000	190 000
0.105	216 000	248 000	0.500	156 000	180 000
0.120	210 000	241 000	0.562	152 000	176 000
0.135	206 000	237 000	0.625	147 000	170 000

Standard Specifications for Carbon-Steel Valve Spring Quality Wire (A 230 – 41):

Table I.—Delete from the table the column specifying minimum elongation requirements.

Section 8.—Revise to read as follows: "A 10-in. specimen twisted seven times forward and reversed until failure shall show a square break normal to the axis of the wire without splits or cracks."

Table II.—Delete Table II.

Section 12(b).—Add an additional sentence to read as follows: "This examination may be made using a binocular microscope with a magnification of 10×."

Standard Specifications for Chromium-Vanadium Steel Valve Spring Quality Wire (A 232 – 41):

Section 2(b).—Revise to read as follows by the addition of the italicized words: "(*b*) The wire shall be *either* in the annealed and cold drawn *or oil tempered* condition, as specified by the purchaser."

Section 7.—Revise to read as follows:

> 7. (*a*) *Annealed and Cold Drawn.*—When in the annealed and cold drawn condition, the wire shall have been given a sufficient amount of final cold working to meet the purchaser's coiling requirements and shall be in a suitable condition to respond properly to heat treatment. In special cases the hardness, if desired, shall be stated in the purchase order.

TABLE X.—TENSILE REQUIREMENTS.

Diameter, in.[a]	Tensile Strength, psi.	
	Min.	Max.
0.032 and under	295 000	315 000
0.041	285 000	305 000
0.054	275 000	295 000
0.062	265 000	285 000
0.080	255 000	275 000
0.105	245 000	265 000
0.135	235 000	255 000
0.162	225 000	245 000
0.192	220 000	240 000
0.244	210 000	230 000
0.283	205 000	225 000
0.437	195 000	215 000

[a] Tensile strength values for intermediate sizes may be interpolated.

> (*b*) *Oil Tempered.*—When purchased in the oil tempered condition, the tensile strength of the wire shall conform to the requirements prescribed in Table I (accompanying Table X) for the various sizes, unless otherwise specified. The reduction of area shall be not less than 45 per cent for sizes 0.156 in. and smaller in diameter and not less than 40 per cent for sizes larger than 0.156 in. in diameter.

New Table.—Add a new Table I as shown in the accompanying Table X.

Section 9.—Revise to read as follows:

> 9. Transverse sections of the wire mounted in plastic, polished, and microetched, shall be examined at a magnification of 100 diameters and the surface of the wire shall exhibit no ferritic area. To reveal the decarburization more accurately in the untempered wire, the specimen shall be hardened and tempered before microscopic examination. Prior to hardening the specimen shall be filed flat on one side enough to reduce the diameter at least 20 per cent. The subsequently mounted specimen shall show the flattened section, as well as the original wire edge. Any decarburization on this flattened section shall necessitate a new specimen for examination.

New Section.—Add a new Section 11 to read as follows, renumbering the subsequent sections accordingly:

> 11. *Workmanship.*—(*a*) The wire shall be uniform in quality and temper and shall not be wavy or crooked.
>
> (*b*) Each bundle shall be one continuous length or wire, properly coiled, and firmly tied.

Standard Specifications for Carbon-Steel Forgings for Locomotives and Cars (A 236 – 46):

Section 3.—Revise to read as follows by deletion of the word in parenthess: "3. A sufficient discard shall be made from each ingot to secure freedom from (injurious) piping and undue segregation."

Section 4.—Revise the first sentence to read as follows by the addition of the italicized word and the deletion of those in parentheses: "*Approximately* (at least) 20 per cent of the forgings shall have prolongations for testing, unless otherwise specified."

Section 6.—In Paragraph (*c*), revise the first sentence to read as follows by the deletion of the word in parentheses: "After forging and after reheating for heat treatment, the forgings shall be cooled (evenly) to a temperature below the critical range."

Revise Paragraph (*d*) to read as follows by the deletion of the word in brackets: "(*d*) *Heating for Heat Treatment.*—The forgings shall be reheated [evenly] to the proper temperature and held at that temperature for a sufficient time to effect the desired change in crystalline (grain) structure."

In Paragraph (*e*), revise the first sentence to read as follows by the deletion of the words in parentheses: "The forgings shall be reheated to and held at the proper temperature and then be allowed to cool slowly (and evenly)."

Table II.—In the first column, revise the class D listing to read, "D (Annealed or Normalized)^b."

Section 10 (*b*).—Revise to read as follows:

> (*b*) The size classification of the forgings shall be determined as follows:
>
> (1) *When Treated After Forging and Prior to Machining.*—When treated after forging and prior to machining the size classification shall be determined by the nominal diameter of the prolongation of the solid forging or the wall thickness of the bored forging.
>
> (2) *When Treated After Machining.*—When treated after machining the size classification shall be determined by the nominal diameter of the solid rough turned forging or the wall thickness of the bored rough turned forging.

Section 12.—Revise the introductory paragraph to read as follows by the addition of the italicized words: "When specified *on the order*, the following microscopic examination of class F forgings shall be made."

In Paragraph (*c*), revise the first sentence to read as follows by the deletion of the words in brackets: "The whole of the transverse section shall show uniform, [well broken up,] fine-grained structure, and shall conform to the requirements illustrated in the photomicrographs, Class F—Fig. 2 (*a*) or (*b*).

Section 13 (*a*).—Revise the introductory sentence to read as follows by the addition of the italicized words: "Unless otherwise specified by the purchaser *on the order*, tension tests shall be made as follows:"

Section 14 (*b*).—Revise to read as follows by the addition of the italicized word: "(*b*) If, when untreated forgings (class A) are specified, the results of the physical tests of any test lot do not conform to the requirements specified, the manufacturer may normalize or anneal such lot, but not more than three *additional* times, and retests shall be made in accordance with Section 13 (*a*).

Section 15 (*a*).—Revise the second sentence to read as follows by the addition of the italicized word: "When centered, *standard* 60-deg. centers with clearance for lathe center points shall be bored."

Section 19 (*a*).—Revise to read as follows by the addition of the italicized words: "(*a*) Unless otherwise specified *on the order*, any rejection based on tests made in accordance with Section 9 shall be reported to the manufacturer within five working days from the receipt of samples by the purchaser."

Standard Specifications for Alloy-Steel Forgings for Locomotives and Cars (A 238 – 46):

Section 3.—Revise to read as indicated above for Section 3 in Specifications A 236.

Section 4.—Revise to read as indicated above for Section 4 in Specifications A 236.

Section 5.—Revise the title to read as follows by the deletion of the words in brackets: "Boring [and Turning]."

Delete the present Paragraph (*d*).

Table I.—In Table I, add figures under the heading "Wall Thickness of Bored Forgings," for Class A, B, and C forgings as follows:

	Wall Thickness of Bored Forgings	
	Over	Not Over
A (Normalized and Tempered)	..	4
	4	8
B (Normalized and Tempered)	..	2½
	2½	4½
	4½	6½
	6½	8
C (Normalized and Tempered)	..	2½
	2½	4½
	4½	6½
	6½	8

Delete the size "10 in." from the first column and "20 in." from the second column, class D and E forgings.

Section 11.—Delete this section renumbering the subsequent sections accordingly.

Section 12.—Revise Paragraph (*a*) to read as follows by the deletion of the words in brackets: "(*a*) Tension test specimens [and bend test specimens when specified] shall be taken from the test prolongations, except as specified in Paragraph (*b*)."

Delete Paragraph (*e*).

Section 13.—Revise the introductory paragraph to read as follows by the addition of the italicized words: "When specified *on the order*, the following microscopic examination of classes A, B, and C forgings shall be made:"

Delete Paragraph (*d*).

Section 14.—Delete this section, renumbering the subsequent sections accordingly.

Section 15.—Delete Paragraph (*b*), relettering the subsequent paragraphs accordingly.

Section 17.—Delete Paragraph (*b*).

Supplementary Requirements.—Add the following Supplementary Requirements at the end of the specifications:

S1. One or more of the following supplementary requirements shall apply only when specified by the purchaser. Details of these supplementary requirements shall be agreed upon by the manufacturer and the purchaser.

Turning

S2. The purchaser may specify a preliminary rough machining prior to the heat treating operation.

Chemical Composition

S3. The purchaser may specify that the residual alloy elements, not intentionally added, shall not exceed 0.35 per cent of copper, 0.25 per cent of nickel, 0.20 per cent of chromium and 0.06 per cent of molybdenum.

Microscopic Test

S4. The longitudinal unetched face may be examined for solid non-metallic impurities, and shall show such impurities well scattered over the field.

Macroscopic Test

S5. The prolongation from the largest forging in each heat shall be sawed normal to the axis of the forging and shall then be split longitudinally. The transverse and longitudinal face shall be etched for macroscopic examination.

Number of Tests

S6. The purchaser may specify one unetched microscopic test and one etched macroscopic test from the largest sized forging in each heat.

Specifications for Welded Alloyed Open-Hearth Iron Pipe (A 253 – 46):

Section 8.—Revise to read as indicated above for Section 8 in Specifications A 53.

Tables II and III.—Delete the present Tables II and III.

New Tables.—Add new Tables II, III, and IV as shown in the accompanying Tables III, IV, and V with the following exceptions:

In new Table II delete grade B and footnote[f]. In new Table III delete grade B. In new Table IV delete grade B and footnote[g].

Section 15.—In Paragraph (*a*) delete the last sentence. Revise Paragraphs (*b*) and (*c*) to read as indicated above for Section 15 (*b*) and (*c*) in Specifications A 53.

Footnote 4.—Revise footnote 4 to read as indicated above for footnote 5 in Specifications A 53.

New Appendix.—Add a new Appendix II to read as indicated above for the new Appendix IV in Specifications A 53.

REPORT OF COMMITTEE A-2*

ON

WROUGHT IRON

Since its last two previous reports to the Society in 1944 and 1945, the latter of which was an informal progress report, the committee has not held any meetings. It has, however, reviewed and considered by correspondence the standards under its jurisdiction. This review has resulted in the recommendations presented in this report.

The committee now consists of 42 members, of whom 13 are classed as producers, 23 as consumers, and 6 as general interest members.

I. ADOPTION OF TENTATIVE REVISION AS STANDARD

In August, 1944, Committee A-2 presented to the Society through the Standards Committee a tentative revision of the Standard Specifications for Wrought Iron Plates (A 42 – 39). This revision provides for adding the following sentence at the end of Section 5 (*f*), which paragraph makes provisions for certain modifications in the tensile requirements: "For material over $\frac{3}{4}$-in. in thickness, the minimum yield point shall not be less than one half the tensile strength." The committee now recommends the adoption as standard of this tentative revision and accordingly asks that this recommendation be approved at the Annual Meeting for submission to letter ballot of the Society.

II. REAFFIRMATION OF STANDARDS

The following eleven standards have been published for six years or longer without revision, but are considered by the committee to be in accord with present practice:

Standard Specifications for:

Refined Iron Bars (A 41 – 36)
Iron and Steel Chain (A 56 – 39)
Wrought Iron Rolled or Forged Blooms and Forgings (A 73 – 39)
Staybolt Wrought Iron, Solid (A 84 – 39)
Common Iron Bars (A 85 – 39)
Staybolt Wrought Iron, Hollow-Rolled (A 86 – 39)
Wrought Iron Rivets and Rivet Rounds (A 152 – 39)
Uncoated Wrought Iron Sheets (A 162 – 39)
Zinc-Coated (Galvanized) Wrought Iron Sheets (A 163 – 39)
Rolled Wrought Iron Shapes and Bars (A 207 – 39)

Standard Definitions of:

Terms Relating to Wrought Iron (A 81 – 33)

The recommendations in this report have been submitted to letter ballot[1] of the committee. The results of the vote will be reported at the Annual Meeting of the Society.

This report has been submitted to letter ballot of the committee, which consists of 42 members; 32 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. M. STEMBEL,
Vice-Chairman.

JAMES ASTON,
Secretary.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

REPORT OF COMMITTEE A-3*

ON

CAST IRON

Since the 1946 Annual Meeting of the Society, Committee A-3 on Cast Iron has held one meeting in Philadelphia, Pa., on February 26, 1947. Thirteen new members and replacements were confirmed and three resignations accepted. The total membership of the committee is now 131.

Considerable time at the Spring Meeting was devoted to discussion of means by which Committee A-3 could expand its activities both in cooperation with other associations interested in cast iron and in furthering cast iron research. A representative of Committee A-3 was designated as the committee representative on the American Foundrymen's Assn. Committees on Chill Control and on Test Bar Development. To facilitate investigation of new research projects, particularly in the field of stress analysis using SR strain gages, the chairman of Committee A-3 appointed a special Research Advisory Group headed by J. S. Vanick.

A history of cast iron specifications and compositions for high-temperature use has been prepared by the chairman of Subcommittee XXII with the view to formulating new tentative specifications for gray iron castings of limited life for temperatures up to 1400 F. in nonpressure service. A meeting of this subcommittee was held on April 29, 1947, in Detroit, Mich., to consider a preliminary draft, and a second meeting will be held during the 1947 Annual Meeting of the Society. Questions have arisen about interpretation of the Standard Specifications for Gray Iron Castings (A 48 – 46) which will necessitate a meeting of Subcommittee VI at the Annual Meeting.

I. Adoption of Tentatives as Standard

The committee is recommending for adoption as standard the following five tentatives:

Tentative Specifications for:

Automotive Gray Iron Castings (A 159 – 44 T),

Lightweight and Thin-Sectioned Gray Iron Castings (A 190 – 44 T),

Tentative Definitions of:

Terms Relating to Cast Iron (A 196 – 46 T)

Tentative Recommended Practice for:

Evaluating Microstructure of Graphite in Gray Iron (A 247 – 41 T), and

Torsion Tests of Cast Iron (A 260 – 42 T), with minor editorial changes.

II. Continuation of Tentatives Without Revision

The committee recommends that the Tentative Specifications for Foundry Pig Iron (A 43 - 45 T) and Gray Iron Castings for Pressure-Containing Parts for Temperatures up to 650 F. (A 278 - 44 T) be continued as tentative, in order to gain wider experience with their use before recommending their adoption as standard. Subcommittees I and XXII who are responsible for these specifications, will hold meetings in June.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

The recommendations in this report have been submitted to letter ballot[1] of the committee, the results of which will be reported at the Annual Meeting.

This report has been submitted to letter ballot of the committee which consists of 131 members; 98 members returned their ballots, of whom 96 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. T. MacKenzie,
Chairman.
E. R. Young,
Vice-Chairman.

C. O. BURGESS,
Secretary.

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

REPORT OF COMMITTEE A-5*
ON
CORROSION OF IRON AND STEEL

Committee A-5 on Corrosion of Iron and Steel held two meetings during the year: One on June 27, 1946, in Buffalo, N. Y., during the Annual Meeting of the Society, and the other on February 27, 1947, in Philadelphia, Pa., during the Spring Group Committee Meetings.

Since the last report, 7 new members have been elected and 4 members have resigned; the total membership of the committee is now 129, of whom 58 are classified as producers, 42 as consumers, and 29 as general interest members.

I. New Tentatives

The committee recommends for publication as tentative the proposed Tentative Specifications for Long Terne Iron or Steel Sheets[1] and the proposed Tentative Method of Test for Weight and Composition of Coating on Long Terne Sheets by the Triple Spot Test.[1]

During the war a draft was prepared of specifications for lead-coated sheets which were being produced in small quantities as a possible substitute for galvanized sheets, or terne sheets. The development of this product and specifications was stopped when the war ended, and as it was expected that tin would again become available, it was decided to formulate specifications for long terne sheets. The specifications submitted cover requirements for both base metal and coating.

The method of test for weight of coating on long terne sheets replaces methods for this product formerly given in Standard A 91 which were discontinued in 1939 as being outmoded. Changes have been made in the old sulfuric acid stripping method and in the methods for determining iron, lead, and tin in the coatings. Additional stripping methods have been added which are suitable for use when iron, lead, or tin determinations are not to be made.

II. Revision of Tentative

The committee recommends that Table I of the Tentative Specifications for Zinc Coating (Hot-Dip) on Iron and Steel Hardware (A 153 - 42 T)[2] be revised as follows by the addition of the italicized figures and the omission of those in brackets:

		Minimum Weight of Coating, oz. per sq. ft. of surface[a]		Uniformity of Coating, minimum number of 1-min. dips in Preece test[a]
		Average of Specimens Tested[b]	Any Individual Specimen	
Class B.—Rolled, pressed, and forged articles	B-1. $\frac{3}{16}$ in. and over in thickness and over 8 in. in length	2.00	*1.80*[1.85]	6
	B-2. Under $\frac{3}{16}$ in. in thickness and over 8 in. in length . . .	*1.50*[2.00]	*1.25*[1.85]	4
	B-3 8 in. and under in length and any thickness	*1.30*[2.00]	*1.10*[1.85]	4
Class C.—Bolts and drive screws (over $\frac{3}{8}$ in. in diameter) and similar articles		1.25	*1.00*[1.15]	4
Class D—Screws, stove bolts, and bolts ($\frac{3}{8}$ in. and under in diameter), rivets, nails, and similar articles		*1.00*[0.90]	*0.85*[0.80]	4

[a] In the case of long pieces, such as anchor rods and similar articles over 5 ft. in length, the weight of coating and uniformity test shall be the average of the determinations made at each end and the middle of the article. In the case of composite pieces, each part shall be tested separately as they may fall in different classifications.

[b] The number of specimens to be tested per order shall be agreed upon, at the time of purchase, by the manufacturer and the purchaser.

Note.—Length of the piece, stated in classes B-1, B-2, and B-3, refers to the overall dimension and not to its developed length.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] These specifications and method were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-A.

[2] 1946 Book of A.S.T.M. Standards, Part I-A.

No change is proposed in the number of dips in the Preece test. The dividing line between classes C and D has been changed. Class C in the present specifications includes articles $\frac{3}{8}$ in. in diameter. The footnote in the present specifications has been omitted and a new footnote added.

These changes are recommended in order that the specifications will call for material which producers can furnish, and are based on analysis of a large number of test specimens. The changes were proposed a year ago but were not recommended at that time because of the reluctance of consumer members to make a downward revision in requirements but are brought forward now on the basis of the tests referred to.

III. REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revision of the Standard Specifications for Zinc (Hot-Galvanized) Coatings on Structural Steel Shapes, Plates, and Bars, and Their Products (A 123 – 33)[2] and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these modifications may be referred to letter ballot of the Society:

Section 5 (a).—Change from its present form: namely,

(*a*) The finished material shall not be injuriously embrittled.[a]

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[a] See Recommended Practice for Safeguarding Against Embrittlement of Hot-Galvanized Structural Steel Products and Procedure for Detecting Embrittlement (A.S.-T.M. Designation: A 143).

to read as follows:

(*a*) Necessary precautions to fabricate properly and prepare the material for galvanizing to prevent embrittlement are described in the Recommended Practice for Safeguarding Against Embrittlement of Hot-Galvanized Structural Steel Products and Procedure for Detecting Embrittlement (A.S.T.M. Designation: A 143). Material found by these tests to be embrittled shall be rejected.

Section 9.—Delete the last sentence which reads: "The coating shall not interfere with the intended use of the material," and substitute the following: "Globules or heavy deposits of zinc which will interfere with the intended use of the material will not be permitted."

These changes do not change the intent of the specifications. The changes in Section 5 (*a*) and (*g*) will clarify the intent of the specifications.

IV. CONTINUATION OF TENTATIVES WITHOUT REVISION

The committee recommends the continuance of the following tentatives without change:

Tentative Specifications for Electrodeposited Coatings of Zinc on Steel (A 164 – 40 T). Committee A-5 has proposed the transfer of Specifications A 164 to Committee B-8 which now sponsors all specifications for electrodeposited metallic coatings. The weights of coatings covered by Specifications A 164 overlap weights of coatings called for in present product specifications sponsored by Committee A-5. Committee A-5 desires, however, to retain control over product specifications. Although A 164 is a specification for coating only, a satisfactory basis on which to make the transfer has not been reached. In addition, revisions are needed in the sampling requirements.

Tentative Methods of Test for Local Thickness of Electrodeposited Coatings (A 219 – 45 T). This method is the joint responsibility of Committees B-8 and A-5. Both committees concur in recommending that these methods remain tentative.

The recommendations appearing in this report have been submitted to letter ballot[3] of the committee, the results of which will be reported at the Annual Meeting.

REORGANIZATION OF SUBCOMMITTEES

Committee A-5 has revised the setup of its subcommittees largely in order to eliminate the concentration of work in two subcommittees. The committee, as formerly constituted, was composed of

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[3] The letter ballot vote on these recommendations was favorable. the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittees III, V, VI, VII, and VIII. In the new organization, former Subcommittees III, VI, and VIII have been eliminated and their duties taken over by Subcommittees XI to XVI, inclusive. No change was made in former Subcommittees V and VII. However, Subcommittee V will shortly become inactive, its actual inactivation awaiting only the submission of a final report. The new organization with the identification of its subcommittees is as follows:

Subcommittees:

V on Immersion Tests, W. J. Jefferies, chairman,

VII on Methods of Testing, R. W. Baker, chairman,

XI on Specifications for Metallic Coated Iron or Steel Sheets and Their Products, E. F. Lundeen, chairman,

XII on Specifications for Metallic Coated Iron or Steel Wire and Its Products, P. E. Chisler, chairman,

XIII on Specifications for Metallic-Coated Iron or Steel Hardware, Structural Shapes, Plates, Bars and Their Products, and Methods for Prevention of Embrittlement of Galvanized Steel, B. J. Barmack, chairman,

XIV on Exposure Testing and Inspection of Black and Metallic Coated Iron or Steel Sheets and Their Products, E. S. Taylerson, chairman,

XV on Exposure Testing and Inspection of Bare and Metallic Coated Iron or Steel Wire and Its Products, A. P. Jahn, chairman, and

XVI on Exposure Testing and Inspection of Bare and Metallic-Coated Hardware, Structural Shapes, Plates, Bars and Their Products, L. J. Waldron, chairman.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VII developed the new method of test for determining weight of coating on long terne sheets, referred to earlier in the report.

Subcommittee XI prepared the new tentative specifications for long terne sheets, referred to earlier in the report.

Subcommittee XII will assume responsibility for the program on wire specifications dropped by the former Subcommittee VI due to the war. Consideration will be given to the need for revision of the specifications for chain-link fence fabric galvanized after weaving (A 117 - 33), and to the specifications for telegraph and telephone line wire (A 111 - 43). It will investigate the need for specifications for chain-link fence fabric galvanized before weaving.

Subcommittee XIII submitted the revision in Table I of Specification A 153 given earlier. In explanation of this revision the following information is presented:

Tentative Specifications A 153 have been studied for several years to enable the specification of reasonable weights of coatings for the multiplicity of articles which comprise pole line hardware. Class B covers rolled, pressed and forged articles, all made of various thicknesses and lengths. Some articles, due to length, are drawn individually or in racks and excess spelter is allowed to drain; other articles, usually 8 in. in length and shorter, are placed in baskets, immersed in molten zinc; and then centrifuged to remove the excess spelter and to provide a smooth coating.

In cooperation with one manufacturer in Chicago, some special samples of rods and bars were made and galvanized by drawn and centrifuged methods, also many samples were taken from daily factory-run of miscellaneous hardware; a total of about 600 pieces were tested. This investigation, which will be described later in the technical press, brought to light several points which are briefly described as follows:

1. Extreme variations in coating weights, under the same controlled conditions of time and temperature, are often due to variations beyond the manufacturer's control.

2. The surface of the base metal is a very important factor. Rough surfaces retain a much heavier coating than steel with a smooth finish. Surface is also affected by the composition of the various steels used in pole line hardware manufacture.

3. The higher carbon steels take heavier coatings. The presence of silicon in percentages of 0.20 to 0.25 is known to result in heavier coating weights.

4. The longer the time of immersion in the molten spelter the heavier will be the coating on low carbon and low-silicon content steels, but only up to a certain point. Long immersions result in coatings that are poorly bonded to the base metal and are likely to flake off, particularly on high-carbon and high-silicon steels. A minimum immersion time of 2½ min. was found to be satisfactory.

5. Centrifuged work is not necessarily inferior to drawn work, but because of its shape and size some material must be run in baskets and will, generally, have a somewhat lighter coating weight. If this material were allowed to drain instead of centrifuging, the coating would be very rough, nonuniform and unacceptable. A rough coating may show a heavy weight per square foot of area, but due to lack of uniformity may not give increased life for that reason.

Subcommittee XIV.—The black sheets at Annapolis, Md., were inspected in the spring and fall of 1946 and six additional failures in the No. 22 gage sheets were found. Two inspections were made by the galvanized sheet inspection group during 1946, the results of which will be given in the 1948 report.

Subcommittee XV.—The report of this subcommittee comprises the Report of Wire Inspection Committee on Field Tests of Wire and Wire Products as appended hereto.[4]

Subcommittee XVI has now been organized for handling hardware exposure tests formerly conducted by Subcommittee VIII and its hardware inspection group. The hardware exposures at State College, Pa., and Sandy Hook, N. J., were inspected on a schedule coinciding with that of the Sheet and Wire Inspection Committees. The only other exposure remaining is at Key West, Fla., which was inspected in April, 1947. Following past practice, the hardware exposure data are scheduled for inclusion in complete form in next year's report. The program of additional exposure tests on hardware parts tentatively planned by Subcommittee VIII at a meeting held in Pittsburgh, Pa., in April, 1946, is now being reviewed and will be presented to the members of Subcommittee XVI for appropriate action.

This report has been submitted to letter ballot of the committee, which consists of 129 members; 93 members returned their ballots, of whom 87 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

T. R. GALLOWAY,
Chairman.

H. F. HORMANN,
Secretary.

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[4] See p. 120.

REPORT OF WIRE INSPECTION COMMITTEE ON FIELD TESTS OF WIRE AND WIRE PRODUCTS*

(COVERS 1945 AND 1946 INSPECTIONS)

NOTE.—Reference to the previously published reports of Subcommittee VIII on Field Tests of Metallic Coatings, of Committee A-5 on Corrosion of Iron and Steel, which appeared in Vol. 39 (1939), Vol. 41 (1941), Vol. 43 (1943), and Vol. 45 (1945), of the *Proceedings*[1] of the American Society for Testing Materials, will be helpful in certain studies of these exposure test results. These earlier reports contain descriptions of the test methods and micrographs and other characterizations of specimens in the test as well as the data assembled in inspections prior to 1945.

This report presents the results of the atmospheric corrosion tests on wire and wire-product specimens after exposure for about ten years at each of eleven locations. These eleven test sites have atmospheres of divers types, including severely industrial, mildly industrial, seacoast, and rural. At each location, more than 900 specimens were exposed. These included short lengths of wire (42 in. long) and wire strand at all locations; farm field fence at nine sites; barbed wire at eight locations; and chain link fence at eight locations. Previous reports show the details of the locations and of the number and nature of the test specimens involved at each location.[1]

The extent of corrosion on specimens is being measured by the following:

1. Visual examination of all specimens at the test locations. At the time of inspection, records are made of the appearance of each specimen. Prior to the time of rusting the specimens are noted as appearing "metallic," "gray," etc. After rusting has begun, estimates are made of the percentage of area rusted on each specimen.

2. Tension tests on the unfabricated wire specimens. These are the specimens exposed in groups of seven comparison samples taken from the same lot of wire and supposedly alike. An attempt is made to remove and test the first specimen of each heptad when it has lost 5 to 10 per cent of its strength, and the seventh specimen when it has lost some 75 per cent of its strength.

3. Loss-of-weight tests on lengths of zinc-coated wire (each about 42 in. long) weighed and measured before exposure.

This year semi-annual inspections have been made at Pittsburgh, Bridgeport, Sandy Hook, and State College by a travelling committee and annual inspections have been made at the other test sites by the university people in charge of the test plots.

Findings from Visual Examination for Corrosion at the Test Plots:

Summaries of the records of inspections through 1946 are shown below in the following condensed tabulations and figure:

Table A shows the corrosion condition of the zinc-coated unfabricated wire specimens after exposure for about ten years at each of eleven sites.

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* Report of Subcommittee XV on Wire Tests (formerly Section on Wire Inspection of Subcommittee VIII on Field Tests of Metallic Coatings) of Committee A-5 on Corrosion of Iron and Steel presented at the Fiftieth Annual Meeting of the American Society for Testing Materials, June 20, 1947.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 157, (1939); Vol 41, p. 101, (1941); Vol. 43, p. 73, (1943); and Vol. 45, p. 70, (1945).

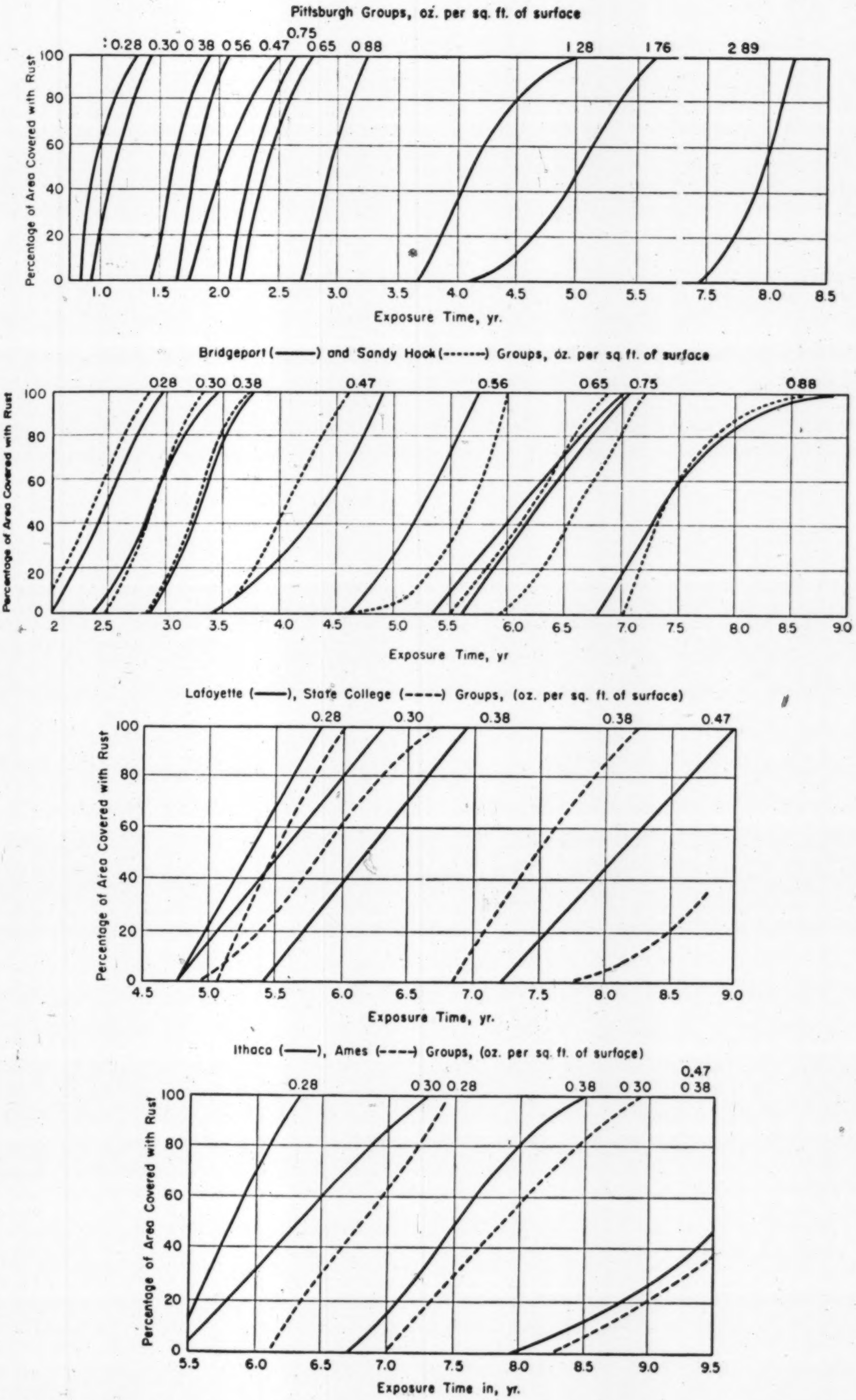

Fig. 1.—Progressive Development of Rust on Zinc-Coated Unfabricated Wire. Coating-weight groups in which all of the samples are rusted.

Exposure time is the average time for samples of any Group to rust to the extent indicated.

TABLE A.—CORROSION CONDITIONS OF ZINC-COATED UNFABRICATED WIRES AFTER APPROXIMATELY TEN YEARS' EXPOSURE.

R = rust; Y = yellowed; G = gray; M = metallic; MG and GY = intermediate states; TrR = trace rust; D = dark to exclusion of better observation; SR and SY = superficial R and Y; R+Y = indistinguishable proportions of R and Y; Gn = green; Br = brown; PHR and PHY = rust or yellowed in pin holes.

Location	Coating Group, oz. per sq. ft. of surface										
	0.20 to 0.30	0.25 to 0.35	0.35 to 0.45	0.45 to 0.55	0.50 to 0.60	0.60 to 0.70	0.70 to 0.85	0.80 to 1.00	1.20 to 1.35	1.60 to 1.85	2.00 or more
Pittsburgh, Pa.	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
Sandy Hook, N. J	100R	100R	100R	100R	100R	100R	100R	90R	40R	3R	G
Bridgeport, Conn.	100R	100R	100R	100R	100R	100R	100R	99R	50R	1R	G
State College, Pa.	100R	100R	100R	76R	35R	6R	1R	GY	GY	G	G
Lafayette, Ind.	100R	100R	100R	100R	60R	60R	40R	10R	G	1R	MG
Ames, Iowa	100R	90R	50R	3R	G	G	G	G	G	G	G
Manhattan, Kans	3R	8R	GY	GY	MG	GY	MG	MG	MG	MG	MG
Ithaca, N. Y.	100R	100R	100R	62R	32R	3R	1R	G	G	M	M
Santa Cruz, Calif.	GY	GY	GY	GY	MG	MG	MG	MG	...	MG	GY
College Station, Tex.	20R	7R	GY	GY	GY	GY	G	G	G	G	G
Davis, Calif.	G	G	G	G	G	G	G	MG	...	MG	MG

TABLE B.—CORROSION CONDITIONS OF UNFABRICATED WIRES CARRYING COATINGS OTHER THAN ZINC AFTER APPROXIMATELY TEN YEARS' EXPOSURE.

R = rust; Y = yellowed; G = gray; M = metallic; MG and GY = intermediate states; TrR = trace rust; D = dark to exclusion of better observation; SR and SY = superficial R and Y; R+Y = indistinguishable proportions of R and Y; Gn = green; Br = brown; PHR and PHY = rust or yellowed in pin holes.

Location	Coating Groups and Corrosion Resistant Groups				
	Copper-Covered Wires[a]	Lead-Coated Wires[b]	12 to 14% Chromium, Cold Drawn	12 to 14% Chromium, Air Quenched	18% Cr, 8% Ni
Pittsburgh, Pa.	D	D70R	DSR	D	D
Sandy Hook, N. J.	Gn	100R+Y	Y	Y	GY
Bridgeport, Conn.	Gn	10R+Y	GY	GY	MG
State College, Pa.	Gn	90R+Y	MG	MG	M
Lafayette, Ind.	Gn	50PHR	M	M	M
Ames, Iowa	Br	70PHY	SY	SY	MY
Manhattan, Kans.	Br	15PHY	G	MG	M
Ithaca, N. Y.	Br	5R+Y	SY	M	M
Santa Cruz, Calif.	Br	20SR	60SR	25SR	MG
College Station, Tex.	Br	20R+Y	90R+Y	50R+Y	M
Davis, Calif.	Br	25SR	45SR	20SR	MG

[a] Weight of coating = 7 to 9 oz. per square foot of surface.
[b] Weight of coating = 1.4 to 1.9 oz. per square foot of surface.

TABLE C.—CORROSION CONDITIONS OF FARM FIELD FENCE AFTER APPROXIMATELY TEN YEARS' EXPOSURE.

R = rust; Y = yellowed; R+Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; TrR = trace rust; PHR, PHY = pinhole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown.

Location	Coating Group, oz. zinc per sq. ft. of surface							Copper Covered[a]	Lead Coated[b]	Cr-Ni Steel
	0.20 to 0.30	0.25 to 0.35	0.35 to 0.45	0.45 to 0.55	0.50 to 0.60	0.60 to 0.70	0.70 or more			
State College, Pa.	100R	95R	75R	20R	10R	5R	G	Gn	30R+Y	MG
Lafayette, Ind.	100R	95R	90R	90R	70R	40R	5R	Gn	15PHR	M
Ames, Iowa	100R	50R	30R	10R	GY	GY	G	Br	50PHY	SY
Manhattan, Kans.	10R	10R	GY	GY	G	MG	MG	Br	15PHR	MG
Ithaca, N. Y.	95R	90R	80R	40R	20R	5R	M	Br	5R	MG
Santa Cruz, Calif.	GY	GY	GY	GY	GY	GY	MG	Br	40SR	SR
College Station, Tex.	15R	15R	GY	TrR	GY	2R	G	Br	5R	SR
Davis, Calif.	G	TrR	G	G	G	MG	MG	Br	50SR	30SR

[a] Weight of coating, 7.42 oz. per sq. ft. of surface.
[b] Weight of coating, 1.48 oz. per sq. ft. of surface.

TABLE D.—CORROSION CONDITIONS OF BARBED WIRE AFTER APPROXIMATELY TEN YEARS' EXPOSURE.

R = rust; Y = yellowed; R+Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; TrR = trace rust; PHR, PHY = pinhole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown.

Location	Coating Group, oz. zinc per sq. ft. of surface							Copper Covered[a]	Lead Coated[b]
	0.20 to 0.30	0.35 to 0.45	0.50 to 0.60	0.70 to 0.85	0.80 to 1.00	1.60 to 1.80	2.00 or more		
State College, Pa.	100R	100R	17R	G	GY	G	G	Gn	...
Lafayette, Ind.	100R	100R	60R	7R	G	G	G	Gn	10R
Ames, Iowa	90R	35R	G	G	G	G	G	Br	60PHY
Manhattan, Kans.	GY	GY	G	MG	MG	MG	MG	Br	10PHY
Ithaca, N. Y.	100R	90R	15R	1R	G	MG	M	Gn	5R
Santa Cruz, Calif.	TrR	G	G	MG	G	G	MG	Br	25SR
College Station, Tex.	7R	2R	1R	G	G	G	G	Br	25R+Y
Davis, Calif.	TrR	G	G	MG	MG	MG	MG	Br	25SR

[a] Weight of coating, 3.97 oz. per sq. ft. of surface.
[b] Weight of coating, 1.83 oz. per sq. ft. of surface.

TABLE E.—CORROSION CONDITIONS OF CHAIN LINK FENCE AFTER APPROXIMATELY TEN YEARS' EXPOSURE.

R = rust; Y = yellowed; R+Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; TrR = trace rust; PHR, PHY = pinhole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown; D = dark (dirt or soot) to the exclusion of a better observation.

Location	Coating Group, oz. zinc per sq. ft. of surface					Copper Covered[c]	Lead Coated[d]	Corrosion Resistant
	0.29 to 0.52[a]	1.13 to 1.22[a]	2.09 to 2.92[a]	1.40 to 1.97[b]	4.21[b] or more			
Pittsburgh, Pa.	100R	100R	100R	100R	50R	...	35R	D
Sandy Hook, N. J.	100R	60R	G	30R	GY	Gn	60R+Y	M
Bridgeport, Conn.	100R	25R	G	30R	GY	Gn	Y	MG
State College, Pa.	80R	GY	G	G	GY	Gn	90R+Y	M
Lafayette, Ind.	95R	G	MG	G	MG	Gn	40PHR	M
Ithaca, N. Y.	90R	G	MG	G	M	Br	5R	M
Santa Cruz, Calif.	GY	MG	MG	MG	MG	Br	20SR	MG
Davis, Calif.	3R	MG	MG	MG	MG	Br	35SR	MG

[a] Coated before weaving.
[b] Coated after weaving.
[c] Weight of coating, 8.52 oz. per sq. ft. of surface.
[d] Weight of coating, 1.75 oz. per sq. ft. of surface.

TABLE F.—CORROSION CONDITIONS OF WIRE STRAND AFTER APPROXIMATELY TEN YEARS' EXPOSURE.

R = rust; Y = yellowed; R+Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; TrR = trace rust; PHR, PHY = pinhole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown; D = dark (dirt or soot) to the exclusion of a better observation.

Location	Coating Group, oz. zinc per sq. ft. of surface				Lead Coated[a]
	0.69 to 0.93	1.03 to 1.17	1.32 to 1.51	1.71 to 2.47	
Pittsburgh, Pa.	100R	100R	70R	40R	30R
Sandy Hook, N. J.	5R	GY	G	G	90R+Y
Bridgeport, Conn.	5R	GY	G	G	70R+Y
State College, Pa.	G	G	G	G	95R+Y
Lafayette, Ind.	MG	G	MG	MG	35PHR
Ames, Iowa	G	G	G	G	95PHY
Manhattan, Kans.	MG	MG	MG	MG	30Y
Ithaca, N. Y.	MG	MG	MG	M	3R
Santa Cruz, Calif.	G	G	G	G	45SR
College Station, Tex.	G	G	G	G	75PHY
Davis, Calif.	G	G	G	G	45SR

[a] Weight of coating, 1.96 oz. per sq. ft. of surface.

Figure 1 shows the progressive development of rust on zinc-coated unfabricated wire specimens for all groups which have become 100 per cent rusted and for all groups of wire, which while not 100 per cent rusted, show some rust on each specimen in the group.

Table B shows the corrosion condition of unfabricated wire of copper-covered steel, lead-coated steel and corrosion-resistant steel exposed at each of eleven test sites.

Table C shows the corrosion condition of farm field fence exposed at all locations except Pittsburgh, Sandy Hook, and Bridgeport. There are many instances where the corrosion state of the top of the fence is more advanced than that of the bottom of the fence; also, different states of corrosion exist between line and stay wires. These differences are not shown in the summary table, but are shown in the detailed Table III. The corrosion performance of any individual specimen of barbed wire or of farm field fence listed in Tables II and III may be directly compared with the performance of specific unfabricated wire specimens. The Reference Information Table (Plate II) in this respect shows the lot numbers of wires used in fabricating all fence and barbed wire samples.

Table D shows the corrosion condition of barbed wire exposed at all locations except Pittsburgh, Sandy Hook, and Bridgeport.

Table E shows the corrosion condition of chain link fence exposed at all locations except Ames, Manhattan, and College Station.

Table F shows the corrosion condition of wire strand exposed at each of eleven test sites.

The detailed corrosion-inspection records collected in 1945 and 1946 are presented at the end of this report in Table I (Plate I) for unfabricated wire specimens, Table II for barbed wire, Table III for farm field fence, Table IV for chain link fence, and Table V for wire strand. These tables are similar in form to the tables in previous reports.

Findings from Specimens Removed for Tension Tests:

There were originally about 840 tensile strength specimens exposed at each site. The results of tensile strength and elongation tests on the 1292 wires removed prior to 1945 have been published in previous reports. A total of 261 tensile strength specimens were removed from the exposure racks and tested during 1945 and 1946. These wires were divided among the different test sites as shown in Table G. The detailed test results for these 261 wires are shown in Table VI. Most of the specimens removed to date have been of the lighter gage wires because these wires lose tensile strength more rapidly than the heavier gage wires.

Pittsburgh is the most corrosive of the test sites and consequently more specimens have been removed from there than from any other location. In the reports for 1943 and 1945 there were summarized the results of tensile strength tests on uncoated and zinc-coated mild steel wires removed from the Pittsburgh site. Although some additional data have been accumulated for the heavier gage wires at Pittsburgh, the data are not sufficient to warrant a revision of the results previously published. One set of corrosion-resistant steel wires (high chromium and nickel-chromium content) was removed from the Pittsburgh racks in 1945 after an exposure of about 8½ yr. These specimens showed no significant change in tensile strength

TABLE G—NUMBER OF TENSILE STRENGTH SPECIMENS REMOVED FOR TEST.

Exposure Site	Number of Wires Removed in 1945 and 1946				Wires Removed Prior to 1945	Total Removed to Date
	Bare Steel	Zinc-Coated	Lead-Coated	Other Types		
Pittsburgh, Pa.	0	41	0	12	546	599
State College, Pa........	7	10	0	0	88	105
Bridgeport, Conn........	7	81	0	0	169	257
Sandy Hook, N. J........	0	103	0	0	207	310
All Other sites	0	0	0	0	282	282
	14	235	0	12	1292	1553

or in elongation from the original condition at the start of the exposure test.

Sandy Hook and Bridgeport, representing sea coast and mildly industrial sites, are less corrosive sites than Pittsburgh but are more corrosive than any of the other sites. A sufficient number of tensile strength specimens of the uncoated wires were assumed to have started to rust immediately on exposure. In the case of the zinc-coated specimens, the time of exposure since first rust was calculated by subtracting from the total time of exposure the time required for first rust to appear as determined from the records of visual inspections.

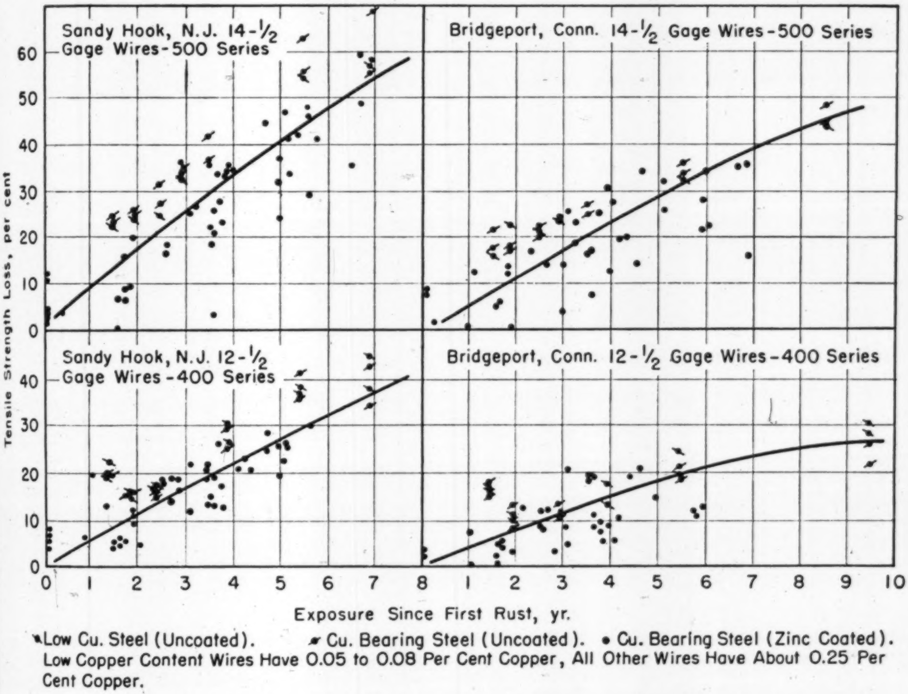

FIG. 2.—Tensile Strength Loss for Periods of Exposure Since First Rust.

lighter gage wires have been removed from these two sites so that a summary of the data can be made. Tables H-1 and H-2 show the results of tensile strength tests on all the Nos. 14½- and 12½-gage uncoated and zinc-coated mild steel wires removed from Sandy Hook and from Bridgeport to date. The table shows for each specimen the exposure time to first rust, the exposure time since first rust, the percentage loss in tensile strength, and the calculated tensile strength loss in per cent per year of exposure since rusting began. All the

In Fig. 2 the data from Tables H-1 and H-2 are plotted to show the loss in tensile strength with different periods of exposure. Through the plotted points a trend line for each wire gage was drawn by eye.

Data from the heavier gage wires at Sandy Hook and at Bridgeport are insufficient for comparisons, and corrosion at the other test sites has not progressed sufficiently to make possible extensive comparisons of tensile strength loss. The accumulated data are, however, sufficient to permit an approximation of the rate of tensile strength loss of the lighter gages of wire at sites of different exposure conditions. For the No. 14½ gage wires the approximate average loss of tensile strength expressed in per cent per year of exposure since first rust, is 14 for Pittsburgh, 10 for Sandy Hook, 6 for Bridgeport, and 4 for State College. For the No. 12½ gage wires the corresponding rates are 10 for Pittsburgh, 6 for Sandy Hook, 3 for Bridgeport, and 2 for State College.

Findings from Weight-Loss Specimens:

At the start of the test 144 wires (16 alike of each of 9 wire lots) were exposed at Pittsburgh, at Sandy Hook, and at State College. The test procedure plan was to remove the specimens one fourth at a time from each location, taking the last quarter when the coating was nearly gone but before rusting started. These tests have now been completed. The last of the specimens

TABLE H-1.—LOSS OF TENSILE STRENGTH SHOWN BY STEEL WIRES AFTER VARIOUS PERIODS OF EXPOSURE, SANDY HOOK, N. J.—500 SERIES (14½ GAGE) AND 400 SERIES (12½ GAGE) UNFABRICATED WIRES.

Wire Lot	Exposure Until First Rust, yr.	First Test			Second Test			Third Test			Fourth Test			Fifth Test			Sixth Test			Seventh Test		
		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss	
			per cent	per cent per year		per cent	per cent per year		per cent	per cent per year		per cent	per cent per year		per cent	per cent per year		per cent	per cent per year		per cent	per cent per year
No. 501[a]	0	1.44	22.0	15	1.90	24.0	13	2.42	25.0	10	2.90	32.0	11	3.45	36.0	11	5.45	54.0	10	6.90	58.0	9
No. 506	0	1.44	25.0	17	1.90	25.0	13	2.42	27.9	11	2.90	32.8	11	3.45	36.5	11	5.45	55.8	10	6.90	56.8	9
No. 507	0	1.44	22.7	16	1.90	26.0	14	2.42	31.1	13	2.90	36.2	12	3.45	42.0	12	5.45	63.0	12	6.90	68.9	10
No. 510	1.75	0	2.0	0	0.15	−2.0	0	1.70	16.0	9	3.70	28.0	8	5.15	41.1	8	6.70	49.0	7	...	...	...
No. 511	1.75	0	12.0	0	0.15	−1.7	0	1.70	7.0	4	3.70	33.3	9	5.15	47.4	9	6.70	59.6	9	...	...	...
No. 512	1.55	0	10.7	0	0.35	4.5	13	1.90	20.0	11	3.90	35.4	9	5.35	41.8	9	6.90	55.4	8	...	...	...
No. 520	2.70	0	3.5	0	1.75	8.9	5	3.75	23.2	6	5.20	33.9	7	5.75	41.1	7	...	...	...	...	...	...
No. 521	2.90	0	3.5	0	1.55	0	0	3.55	20.4	6	5.00	31.5	6	5.55	46.3	8	...	...	...	...	...	...
No. 522	2.90	0	2.0	0	1.55	7.0	5	3.55	26.0	7	5.00	37.0	7	5.55	48.0	9	...	...	...	...	...	...
No. 530	2.90	3.55	3.2	1	5.00	24.2	5	5.55	29.0	5	6.50	35.5	5	...	...	...	...	...	...	...	...	...
No. 531	4.70	1.75	9.0	5	3.20	27.0	8	3.75	33.0	9	4.70	45.0	7	...	...	...	...	...	...	...	...	...
No. 540	5.90	2.55	18.3	7	3.50	21.7	6	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 541	5.40	3.05	25.7	8	4.00	34.3	9	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 542	5.50	2.95	35.2	12	3.90	34.3	9	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 543	5.90	2.55	16.7	6	3.50	18.3	5	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 400[a]	0	1.44	22.0	13	1.90	14.5	8	2.42	15.6	6	3.90	26.0	7	5.45	36.5	7	6.90	37.7	5	...	...	...
No. 401[a]	0	1.44	18.7	12	1.90	15.5	8	2.42	16.7	7	3.90	30.0	8	5.45	37.8	7	6.90	44.4	6	...	...	...
No. 405	0	1.44	19.6	14	1.90	15.5	8	2.42	15.5	6	3.90	29.9	8	5.45	41.2	8	6.90	42.3	6	...	...	...
No. 406	0	1.44	19.0	13	1.90	15.0	8	2.42	14.9	6	3.90	25.5	7	5.45	35.1	6	6.90	34.0	5	...	...	...
No. 410	1.80	0	3.5	0	1.65	5.3	3	3.10	21.9	7	3.65	18.4	5	5.10	25.5	5	...	...	...	...	...	...
No. 411	1.85	0	−5.7	0	1.60	4.9	3	3.05	11.4	4	3.60	14.6	4	5.05	21.9	4	...	...	...	...	...	...
No. 412	2.50	0	5.0	0	0.95	6.0	6	2.40	...	...	2.95	...	...	4.40	20.5	5	...	...	...	...	...	...
No. 421	2.80	0	6.5	0	1.65	4.3	3	3.65	12.8	4	5.10	25.5	5	...	...	...	...	...	...	...	...	...
No. 422	2.90	0	6.0	0	1.55	4.0	3	3.55	13.0	4	5.00	19.0	4	...	...	...	...	...	...	...	...	...
No. 423	2.75	0	7.5	0	1.70	5.0	3	3.70	12.5	3	...	...	...	...	...	...	...	...	...	...	...	...
No. 430	4.40	2.05	4.5	2	3.50	21.2	6	4.05	20.5	5	5.00	25.7	5	...	...	...	...	...	...	...	...	...
No. 431	3.70	2.75	13.1	5	4.20	22.7	5	4.75	24.4	5	5.70	30.0	6	...	...	...	...	...	...	...	...	...
No. 432	6.50	0	−10.0	0	1.40	12.7	9	1.95	10.8	5	2.90	16.0	5	...	...	...	...	...	...	...	...	...
No. 440	4.70	3.75	26.3	7	4.70	28.0	6	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 441	6.50	1.95	11.5	6	2.90	18.3	6	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 442	7.40	1.05	19.2	18	2.00	9.6	5	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 443	5.90	2.55	18.0	7	3.50	20.7	6	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 444	5.70	2.75	18.4	7	3.70	17.5	5	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 495	5.90	2.55	17.3	7	3.50	18.0	5	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...

[a] Low copper content wires 0.05 to 0.08 per cent copper. All other wires have approximately 0.025 per cent copper.

were removed from State College in 1945, and the results obtained from these specimens are shown in Table VII. Similar detailed data from the earlier removals from Pittsburgh, Sandy Hook, and State College were reported in 1939, 1941, 1943, and 1945.

TABLE H-2.—LOSS OF TENSILE STRENGTH SHOWN BY STEEL WIRES AFTER VARIOUS PERIODS OF EXPOSURE, BRIDGEPORT, CONN.—500 SERIES (14½ GAGE) AND 400 SERIES (12½ GAGE) UNFABRICATED WIRES.

Wire Lot	Exposure Until First Rust, yr.	First Test			Second Test			Third Test			Fourth Test			Fifth Test			Sixth Test			Seventh Test		
		Exposure Since 1R, yr.	Tensile Str. Loss per cent	Tensile Str. Loss per cent per year	Exposure Since 1R, yr.	Tensile Str. Loss per cent	Tensile Str. Loss per cent per year	Exposure Since 1R, yr.	Tensile Str. Loss per cent	Tensile Str. Loss per cent per year	Exposure Since 1R, yr.	Tensile Str. Loss per cent	Tensile Str. Loss per cent per year	Exposure Since 1R, yr.	Tensile Str. Loss per cent	Tensile Str. Loss per cent per year	Exposure Since 1R, yr.	Tensile Str. Loss per cent	Tensile Str. Loss per cent per year	Exposure Since 1R, yr.	Tensile Str. Loss per cent	Tensile Str. Loss per cent per year
No. 501[a].	0	1.45	16.0	11	1.91	23.0	12	2.42	21.0	9	2.91	24.0	8	5.45	33.0	6	8.45	44.0	5	...	...	...
No. 506..	0	1.45	21.2	15	1.91	17.4	9	2.42	22.1	9	3.45	25.0	7	5.45	33.6	6	8.45	45.2	5	...	...	...
No. 507	0	1.45	17.6	12	1.91	17.6	9	2.42	20.2	8	3.45	26.9	8	5.45	36.1	6	8.45	48.6	6	...	...	...
No. 510	2.50	0	−2.0	0	0.06	−6.0	0	1.60	5.0	3	3.44	16.0	5	5.95	28.0	5	...	...	...	...	...	...
No. 511..	1.80	0	8.8	0	0.11	−0.4	0	1.65	5.3	3	4.14	19.3	5	6.65	35.1	5	...	...	...	...	...	...
No. 512..	1.65	0	7.6	0	0.26	1.7	0	1.80	13.8	8	4.29	20.0	5	6.85	35.4	5	...	...	...	...	...	...
No. 520..	2.50	1.95	0	0	3.95	12.5	3	5.95	21.4	4	6.90	16.1	2	...	...	...	...	...	...	...	...	...
No. 521	3.45	1.00	−3.0	0	3.00	3.7	1	5.05	25.9	5	6.00	22.2	4	...	...	...	...	...	...	...	...	...
No. 522	3.45	1.00	1.0	0	3.00	14.0	5	5.05	32.0	6	6.00	34.0	6	...	...	...	...	...	...	...	...	...
No. 530..	5.40	1.05	12.9	12	2.50	19.3	8	3.05	25.2	8	4.00	27.5	7	...	...	...	...	...	...	...	...	...
No. 531..	4.70	1.75	12.0	7	3.20	23.0	7	3.75	25.0	7	4.70	34.0	7	...	...	...	...	...	...	...	...	...
No. 540..	6.20	2.25	16.7	7	3.20	18.4	6	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 541..	4.90	3.55	7.1	2	4.50	14.3	3	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 542..	5.50	2.95	23.6	8	3.90	30.4	8	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 543..	5.90	2.55	13.3	5	3.50	16.7	5	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 400[a]	0	1.45	16.5	12	1.91	10.5	6	3.91	13.5	4	5.45	19.8	4	9.42	28.1	3	...	...	...	...	...	...
No. 401[a]	0	1.45	15.5	11	1.91	11.0	6	3.91	17.8	5	5.45	24.4	4	9.42	30.0	3	...	...	...	...	...	...
No. 405..	0	1.45	17.5	12	1.91	13.5	7	2.91	13.4	5	5.45	20.6	4	9.42	25.8	3	...	...	...	...	...	...
No. 406 .	0	1.45	15.0	11	1.91	8.5	4	2.91	10.7	4	5.45	18.1	3	9.42	21.3	2	...	...	...	...	...	...
No. 410..	1.80	0	3.5	0	0.11	−5.3	0	1.65	3.5	2	4.14	10.5	3	...	...	...	...	...	...	...	...	...
No. 411..	1.90	0	2.5	0	0	−11.4	0	1.55	1.6	1	4.04	5.7	1	...	...	...	...	...	...	...	...	...
No. 412	2.50	0	0	0	0	0	0	1.00	7.2	7	3.44	...	...	...	...	...	...	...	...	...	...	...
No. 421	2.70	1.75	3.2	2	3.75	9.6	3	5.75	11.7	2	...	...	...	...	...	...	...	...	...	...	...	...
No. 422	2.70	1.75	5.0	3	3.75	7.0	2	5.75	11.0	2	...	...	...	...	...	...	...	...	...	...	...	...
No. 423	2.50	1.95	2.5	1	3.95	8.7	2	5.95	12.5	2	...	...	...	...	...	...	...	...	...	...	...	...
No. 430..	4.90	1.55	...	...	3.00	8.3	3	3.65	8.3	2	...	...	...	...	...	...	...	...	...	...	...	...
No. 431..	3.50	2.95	10.2	3	4.40	18.7	4	4.95	14.8	3	...	...	...	...	...	...	...	...	...	...	...	...
No. 432..	5.40	1.05	0	0	2.50	8.8	4	3.05	4.9	2	...	...	...	...	...	...	...	...	...	...	...	...
No. 440..	4.80	3.65	11.0	3	4.60	20.3	5	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 441..	5.90	2.55	8.6	3	3.50	18.3	5	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 442..	6.30	2.15	12.8	6	3.10	20.2	6	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 443..	5.80	2.65	12.0	5	3.60	18.6	5	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 444..	5.60	2.85	2.6	1	3.80	4.4	1	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 495..	5.90	2.55	12.0	5	3.50	18.7	5	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...

[a] Low copper content wires 0.05 to 0.08 per cent copper. All other wires have approximately 0.25 per cent copper.

In Table J* are summarized the results of the weight-loss tests. The data from all three sites and for different exposure periods have been reduced to the common comparative unit—loss of zinc coating in ounces per square foot of surface per year of exposure. The over-all average annual loss of coating in ounces per square foot of surface at Pittsburgh is 0.369; at Sandy Hook it is 0.117; and at State College it is 0.060.

There is an indication in the data from the Pittsburgh site that there is a very slight decrease in the rate of coating loss with increasing periods of exposure. However, although the data from the Sandy Hook and State College sites

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* See p. 128

TABLE J.—LOSS OF WEIGHT OF ZINC COATING, OUNCES PER SQUARE FOOT PER YEAR.

Wire Lot	Original Coating Weight, oz. per sq. ft.[d]	Pittsburgh, Pa.[a]					Sandy Hook, N. J.					State College, Pa.				
		1.48 yr.	1.49 yr.	2.45 yr.	2.93 yr.	Avg.	1.44 yr.	2.41 yr.	3.45 yr.	4.44 yr.	Avg.	1.30 yr.	3.31 yr.	6.30 yr.	8.29 yr.	Avg.
GROUP A[b]																
No. 148	0.90	0.343[c]	0.354	0.314	...	0.337	0.114	0.107	0.104	0.105	0.107	0.069	0.058	0.060	0.061	0.061
No. 346	0.88	0.398	0.392	0.364	...	0.385	0.119	0.113	0.112	0.119	0.116	0.062	0.059	0.061	0.064	0.061
No. 542	0.70	0.448	...	...	...	0.448	0.136	0.114	0.118	0.122	0.122	0.077	0.059	0.061	0.067	0.066
GROUP B[b]																
No. 247	0.96	0.349	0.355	0.337	...	0.347	0.118	0.126	0.126	0.121	0.123	0.084	0.063	0.061	0.063	0.068
No. 345	0.84	0.376	0.365	0.329	...	0.357	0.137	0.131	0.127	0.129	0.131	0.084	0.062	0.061	0.067	0.068
No. 541	0.76	0.395	0.374	0.326	...	0.365	0.142	0.139	0.134	0.141	0.139	0.076	0.058	0.060	0.066	0.060
GROUP C[b]																
No. 248	0.81	0.349	0.315	...	...	0.332	0.107	0.095	0.099	0.092	0.098	0.051	0.050	0.049	0.051	0.050
No. 348	1.05	0.359	0.343	0.368	0.328	0.349	0.112	0.099	0.100	0.099	0.102	0.065	0.049	0.050	0.052	0.053
No. 540	0.70	0.428	0.377	...	...	0.402	0.113	0.105	0.106	0.105	0.112	0.074	0.046	0.050	0.050	0.055
ALL GROUPS																
Average	0.84	0.383	0.359	0.340	0.328	0.369	0.124	0.114	0.114	0.115	0.117	0.071	0.056	0.057	0.060	0.060

[a] Blank spaces under Pittsburgh heading indicate no data available because rusting had started prior to removal dates.

[b] The wire lot numbers listed under group A (or B or C) are closely alike as regards base-metal composition and coating characteristics.

[c] Each weight-loss figure is the average weight loss as determined from four specimens of the wire lot.

[d] The zinc-coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

TABLE K.—LOSS OF WEIGHT OF ZINC COATING.

Gage[a] of Wire	Loss of Weight of Zinc Coating, oz. per sq. ft. per yr. (Averages of Like Gages)		
	Pittsburgh	Sandy Hook	State College
6	0.337	0.107	0.061
9	0.341	0.112	0.059
11	0.358	0.114	0.058
14½	0.391	0.123	0.062

[a] Gage 6 wires are 100 series wire lot number, gage 9 wires are 200 series, gage 11 wires are 300 series, and gage No. 14½ wires are 500 series.

TABLE L.—AVERAGES OF COATING-WEIGHT LOSS.

Group[a]	Loss of Weight of Zinc Coating, oz. per sq. ft per yr. (Average of Each Group)		
	Pittsburgh	Sandy Hook	State College
A	0.390	0.115	0.063
B	0.356	0.131	0.065
C	0.361	0.104	0.053

[a] The wire lot numbers in groups A (or B or C) are closely alike as regards base-metal compositon and coating characteristics.

show a higher loss rate for the first determination there is no subsequent steady decrease in the rate of loss with increased exposure periods.

There is a tendency for the lighter gages of wire to lose coating somewhat faster than the heavier gages. This is shown in Table K.

There is no consistent indication that any one of the three groups (A or B or C) loses coating at a more rapid rate than the other two. In Table L are summarized the averages of coating-weight loss in ounces per square foot per year for each group at each of the three sites.

The time required for first rust to appear on samples thus far rusted at Pittsburgh, Sandy Hook, and State College checks reasonably well with the time that would be expected from the data on the weight-loss specimens.

Respectfully submitted on behalf of the Wire Test Inspection Committee,

A. P. JAHN,

Chairman, Subcommittee XV

TABLE I - REPORT OF INSPECTIONS OF UNFABR

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = Gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. R = rust of base metal. PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance, D = dark (dirt or soot) to the exclusion of a better observation. Cr = yellow from chromate treatment. MG = intermediate between M and G. Gy = predominately gray but showing indication of Y. Figures (as 5, 20, 50, 100) are percentages.

Coating Group	Average Coating, oz. per sq.ft.of Surface[a]	Wire	State College, Pa.				Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N.Y.		Santa Cruz, Cal.		College Sta., Tex.		Davis, Cal.	
			8.29 Yr.	8.77 Yr.	9.27 Yr.	9.77 Yr.	8.99 Yr.	9.98 Yr.	8.88 Yr.	9.96 Yr.	8.80 Yr.	9.94 Yr.	9.00 Yr.	9.92 Yr.	8.84 Yr.	9.85 Yr.	8.88 Yr.	9.84 Yr.	8.88 Yr.	9.84 Yr.
0.20	0.27	No. 512	100R	100R	100R	100R	100R	100R	100R	100R	Y	7R	100R	100R	GY	GY	12R	37R	GY	GY
to	0.30	No. 410	100R	100R	100R	100R	100R	100R	100R	100R	Y	7R	100R	100R	GY	GY	12R	71R	GY	GY
0.30	0.28	No. 411	70R	100R	100R	100R	100R	100R	100R	100R	G	G	100R	100R	G	GY	GY	1R	G	G
Avg.	0.29	No. 510	10R	100R	100R	100R	100R	100R	100R	100R	Y	1R	100R	100R	GY	GY	GY	1R	G	G
0.28	0.29	No. 412	10R	100R	100R	100R	100R	100R	100R	100R	Y	1R	100R	100R	GY	GY	GY	GY	G	G
	0.24	No. 511	100R	100R	100R	100R	100R	100R	100R	100R	Y	1R	100R	100R	GY	GY	2R	10R	G	G
0.25	0.25	No. 315	100R	100R	100R	100R	100R	100R	100R	100R	5R	25R	100R	100R	1R	GY	7R	30R	GY	GY
to	0.28	No. 215	100R	100R	100R	100R	100R	100R	100R	100R	10R	20R	100R	100R	GY	GY	4R	12R	GY	GY
0.35	0.27	No. 117	100R	100R	100R	100R	100R	100R	100R	100R	5R	8R	100R	100R	GY	GY	2R	5R	GY	GY
Avg.	0.38	No. 317	45R	99R	100R	100R	100R	100R	G	9R	G	GY	82R	100R	G	G	GY	GY	G	G
0.30	0.36	No. 217	100R	100R	100R	100R	100R	100R	38R	100R	Y	1R	100R	100R	GY	GY	GY	1R	G	G
	0.32	No. 316	100R	100R	100R	100R	100R	100R	40R	100R	Y	GY	100R	100R	GY	GY	GY	GY	G	G
	0.30	No. 216	100R	100R	100R	100R	100R	100R	34R	96R	Y	GY	100R	100R	GY	GY	GY	GY	G	G
	0.25	No. 115	100R	100R	100R	100R	100R	100R	97R	100R	12R	9R	100R	100R	GY	GY	4R	9R	G	G
0.35	0.36	No. 520	100R	100R	100R	100R	100R	100R	79R	100R	G	GY	100R	100R	GY	GY	GY	GY	G	G
to	0.35	No. 422	85R	100R	100R	100R	100R	100R	1R	23R	Y	GY	100R	100R	GY	GY	GY	GY	G	G
0.45	0.31	No. 423	100R	100R	100R	100R	100R	100R	25R	100R	Y	1R	100R	100R	GY	GY	GY	GY	G	G
Avg.	0.44	No. 522	62R	100R	100R	100R	100R	100R	G	24R	G	GY	100R	100R	GY	GY	GY	GY	G	G
0.38	0.37	No. 282	95R	100R	100R	100R	100R	100R	1R	43R	Y	GY	100R	100R	GY	GY	GY	1R	G	G
	0.38	No. 521	77R	100R	100R	100R	100R	100R	2R	12R	G	GY	100R	100R	GY	GY	GY	GY	G	G
	0.47	No. 421	75R	100R	100R	100R	100R	100R	13R	49R	Y	GY	100R	100R	GY	GY	GY	GY	G	G
0.45	0.44	No. 325	2R	40R	60R	95R	100R	100R	G	G	G	GY	6R	96R	GY	GY	GY	GY	G	G
to	0.50	No. 228	10R	95R	100R	100R	99R	100R	G	1R	Y	GY	71R	100R	GY	GY	GY	GY	G	G
0.55	0.54	No. 329	6R	30R	40R	70R	80R	100R	1R	2R	G	GY	7R	43R	GY	GY	GY	1R	G	G
Avg.	0.52	No. 287	Y	10R	10R	40R	80R	100R	G	G	Y	GY	G	18R	GY	GY	GY	GY	G	G
0.47	0.41	No. 125	80R	100R	100R	100R	100R	100R	1R	25R	Y	GY	99R	100R	GY	GY	GY	1R	G	G
	0.54	No. 127	Y	2R	10R	70R	84R	100R	G	G	Y	1R	4R	34R	GY	GY	GY	1R	G	G
	0.38	No. 326	15R	50R	70R	95R	95R	100R	G	2R	G	G	73R	95R	G	G	GY	GY	G	G
	0.45	No. 226	Y	8R	8R	30R	75R	99R	G	G	Y	GY	8R	29R	GY	G	GY	GY	G	G
	0.47	No. 327	1R	10R	15R	80R	90R	100R	G	G	G	G	1R	76R	G	GY	GY	GY	G	G
	0.45	No. 225	1R	30R	30R	80R	65R	99R	G	G	Y	G	1R	33R	G	GY	GY	GY	G	G
0.50	0.61	No. 530	G	G	G	G	G	9R	G	G	MG	MG	MG	G	MG	MG	G	G	MG	MG
to	0.57	No. 432	G	G	G	G	G	1R	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
0.60	0.60	No. 531	Y	3R	3R	35R	90R	100R	G	G	MG	MG	3R	58R	MG	MG	G	G	MG	G
Avg.	0.48	No. 431	10R	99R	100R	100R	100R	100R	G	G	G	GY	90R	100R	GY	G	GY	GY	G	G
0.56	0.54	No. 430	Y	5R	10R	40R	42R	97R	1R	1R	G	G	1R	1R	G	G	GY	GY	G	G
0.60	0.69	No. 338	G	G	G	G	G	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
to	0.68	No. 236	G	G	G	G	G	2R	G	G	MG	GY	M	M	MG	MG	G	G	MG	G
0.70	0.61	No. 335	2R	10R	15R	40R	84R	100R	G	G	MG	MG	1R	23R	MG	MG	G	G	MG	G
Avg.	0.63	No. 237	GY	5R	10R	15R	23R	43R	G	G	MG	MG	1R	3R	MG	MG	G	G	MG	G
0.65	0.74	No. 135	GY	GY	GY	Y	G	26R	G	G	G	G	G	GY	MG	MG	G	G	G	G
	0.57	No. 337	Y	Y	Y	3R	90R	100R	G	G	Y	3R	G	1R	GY	MG	GY	1R	G	G
	0.67	No. 235	Y	Y	Y	Y	G	24R	G	G	GY	GY	G	GY	GY	GY	GY	1R	G	G
	0.65	No. 239	Y	Y	Y	Y	6R	80R	G	G	G	GY	G	1R	GY	GY	GY	1R	G	G
	0.67	No. 138	Y	Y	Y	Y	10R	69R	G	G	Y	GY	G	GY	GY	GY	GY	1R	G	G
	0.64	No. 139	Y	1R	2R	5R	23R	70R	G	G	Y	GY	1R	3R	GY	GY	GY	2R	G	G
	0.66	No. 336	Y	Y	Y	1R	11R	89R	G	G	G	GY	G	GY	GY	GY	GY	GY	G	G
	0.60	No. 238	Y	2R	3R	10R	40R	98R	G	G	G	GY	1R	1R	GY	GY	GY	GY	G	G
0.70	0.70	No. 540	G	G	G	G	G	G	G	G	MG	MG	MG	G	MG	MG	G	G	MG	MG
to	0.72	No. 442	G	G	G	G	G	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	G
0.85	0.79	No. 543	GY	GY	GY	2R	4R	27R	G	G	MG	MG	G	1R	MG	MG	G	G	MG	G
Avg.	0.78	No. 441	GY	GY	GY	1R	G	G	G	1R	MG	MG	G	G	MG	MG	G	G	MG	G
0.75	0.70	No. 542	GY	GY	GY	GY	10R	99R	G	G	MG	MG	G	GY	MG	MG	G	G	MG	G
	0.74	No. 443	GY	GY	GY	GY	G	15R	G	G	MG	MG	G	1R	MG	MG	G	G	MG	MG
	0.74	No. 495	Cr	GY	GY	GY	G	G	G	G	Cr	Cr	G	G	-	-	G	Cr	-	-
	0.76	No. 541	Y	Y	Y	Y	30R	98R	G	G	G	GY	G	GY	GY	GY	GY	GY	G	G
	0.76	No. 440	Y	Y	Y	10R	90R	100R	G	G	Y	GY	G	4R	GY	GY	GY	GY	G	G
	0.87	No. 444	Y	Y	Y	Y	G	48R	G	G	Y	5R	G	GY	GY	GY	G	1R	G	G
0.80	1.05	No. 348	G	G	G	G	G	G	G	G	MG	MG	M	M	G	MG	G	G	MG	MG
to	0.81	No. 248	G	G	G	G	G	G	G	G	MG	MG	M	M	MG	G	G	G	MG	MG
1.00	0.88	No. 347	G	G	G	G	G	G	G	G	M	MG	G	G	MG	MG	G	G	MG	MG
Avg.	0.95	No. 246	GY	GY	G	G	G	1R	G	G	MG	MG	MG	G	MG	MG	G	G	MG	MG
0.88	0.88	No. 346	Y	Y	Y	GY	G	1R	G	G	MG	MG	G	GY	G	G	G	G	MG	MG
	0.81	No. 245	Y	Y	Y	1R	G	16R	G	G	MG	MG	G	GY	MG	G	G	G	MG	G
	0.81	No. 296	Cr	Cr	Cr	Cr	G	G	G	G	Cr	Cr	G	G	-	-	-	-	-	-
	0.90	No. 148	GY	GY	GY	GY	G	3R	G	G	G	G	G	GY	MG	G	G	G	MG	G
	0.85	No. 149	G	G	G	G	3R	13R	G	G	G	MG	G	G	MG	MG	G	G	MG	MG
	0.84	No. 345	Y	Y	Y	Y	9R	71R	G	G	Y	GY	G	GY	GY	G	GY	2R	G	G
	0.96	No. 247	Y	Y	Y	Y	G	6R	G	G	G	MG	G	1R	G	G	GY	1R	G	G
Avg.	1.35	No. 800	G	G	GY	GY	G	G	G	G	MG	MG	G	G	-	-	G	G	-	-
1.28	1.20	No. 801	GY	G	GY	GY	G	G	G	G	MG	MG	G	GY	-	-	G	G	-	-
1.60	2.08	No. 552	G	G	G	G	G	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
to	1.83	No. 451	G	G	G	G	G	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
1.80	1.75	No. 350	G	G	G	G	G	G	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
Avg.	1.69	No. 252	G	G	G	G	G	G	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
1.76	1.65	No. 351	G	G	G	G	G	1R	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
	1.76	No. 250	G	G	G	G	G	G	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	G
	1.69	No. 551	G	G	G	G	G	MG	G	G	M	MG	M	M	MG	G	G	G	MG	MG
	1.50	No. 450	G	G	G	G	G	G	G	G	MG	MG	M	M	G	G	G	G	MG	MG
	1.75	No. 352	G	G	G	G	G	6R	G	G	MG	MG	M	M	G	G	G	G	MG	G
	1.86	No. 251	G	G	G	G	G	MG	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
	1.86	No. 151	G	G	G	G	G	G	G	G	MG	MG	MG	G	MG	MG	G	G	MG	MG
Heavy	2.80	No. 397	G	G	G	G	G	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
Avg.	2.72	No. 298	G	G	G	G	G	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
2.89	3.00	No. 299	G	G	G	G	G	G	G	G	MG	MG	M	M	-	-	G	G	-	-

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[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

PLATE I
PROCEEDINGS, AM. SOC. TESTING MATS.
VOL. 47
REPORT OF COMMITTEE A-5:
WIRE INSPECTION REPORT

UNFABRICATED WIRE SPECIMENS

Abbreviations and symbols used to designate appearance or states of corrosion: L = Metallic, G = grey, Y = yellowed, R = rust of base metal, PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance. D = dark (dirt or soot) to the exclusion of a better observation. MG = intermediate between M and G. GY predominately gray but showing indication of Y. SY* or SR* indicate, respectively: superficial Y and superficial, R, the asterisk denoting a speckled appearance. Figures (as 5, 20, 50, 100) are percentages. R+Y = indistinguishable proportions of R and Y.

	Gage	Wire	Pittsburgh (Brunot Island), Pennsylvania			Sandy Hook, N. J.				Bridgeport, Conn.				State College, Pa.			
			8.47 Yr.	8.94 Yr.	9.44 Yr.	8.44 Yr.	8.92 Yr.	9.42 Yr.	9.92 Yr.	8.44 Yr.	8.92 Yr.	9.42 Yr.	9.92 Yr.	8.29 Yr.	8.77 Yr.	9.27 Yr.	9.77 Yr.
Copper-covered wire	11	No. 021, 7.42-oz.	D	D	D	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn
	9	No. 022, 9.24-oz.	D	D	D	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn
Lead-coated Wires	6	No. 040, 1.40-oz.	D40R	D30R	D40R	100R+Y	90R+Y	100R+Y	100R+Y	1R	1R	5R	5R	80R+Y	55R+Y	100R+Y	100R+Y
	9	No. 041, 1.48-oz.	D40R	D50R	D50R	100R+Y	90R+Y	100R+Y	100R+Y	1R	2R	1R	10R	80R+Y	55R+Y	70R+Y	100R+Y
	11	No. 042, 1.68-oz.	D50R	D80R	D60R	100R+Y	90R+Y	100R+Y	100R+Y	1R	2R	1R	10R	80R+Y	50R+Y	80R+Y	90R+Y
	14½	No. 043, 1.90-oz.	D90R	D90R	D90R	100R+Y	90R+Y	100R+Y	100R+Y	1R	1R	1R	5R	80R+Y	50R+Y	80R+Y	90R+Y
Corrosion-Resistant Uncoated Wires:																	
12-14% Cr., Cold drawn	12	No. 001	DSR	DSR	DSR	Y	YSR	Y	YSR	G	Y	GY	GY	M	M	M	MG
	9	No. 004	DSR	DSR	DSR	Y	YSR	Y	Y	G	Y	GY	GY	M	M	M	MG
	17	No. 005	DSR	DSR	DSR	Y	YSR	Y	Y	G	Y	GY	GY	M	M	M	MG
12-14% Cr., air quenched	13	No. 002	DSR	DSR	D	Y	YSR	Y	Y	G	GY	GY	GY	M	M	M	MG
	10	No. 006	DSR	DSR	D	Y	YSR	Y	Y	G	GY	GY	GY	M	M	M	MG
	18	No. 007	DSR	DSR	D	Y	YSR	Y	Y	G	GY	GY	GY	M	M	M	MG
18% Cr., 8% Ni.	15	No. 003	D	D	D	MG	MG	MG	GY	MG	MG	MG	MG	M	M	M	M
	12	No. 008	D	D	D	MG	MG	MG	GY	MG	MG	MG	MG	M	M	M	M
	20	No. 009	D	D	D	MG	MG	MG	GY	MG	MG	MG	MG	M	M	M	M
	14	No. 015	D	D	D	MG	MG	MG	GY	MG	MG	MG	MG	M	M	M	M

	Gage	Wire	Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N.Y.		Santa Cruz, Calif.		College Sta., Texas		Davis, Calif.	
			8.99 Yr.	9.98 Yr.	8.88 Yr.	9.96 Yr.	8.80 Yr.	9.94 Yr.	9.00 Yr.	9.92 Yr.	8.84 Yr.	9.85 Yr.	8.88 Yr.	9.84 Yr.	8.88 Yr.	9.84 Yr.
Copper-covered wire	11	No. 021, 7.42-oz.	Gn	Gn	Br	Br	Br	Br	B	B	Br	Br	Br	Br	Br	Br
	9	No. 022, 9.24-oz.	Gn	Gn	Br	Br	Br	Br	B	B	Br	Br	Br	Br	Br	Br
Lead-coated Wires	6	No. 040, 1.40-oz.	40PHR	50PHR	34PHY	69PHY	5R*	15PHY	2R	5R+Y	20SR	19SR	14PHY	14PHY	21SR	20SR
	9	No. 041, 1.48-oz.	30PHR	40PHR	44PHY	71PHY	10R	12R	12R	9R+Y	34SR	36SR	32PHY	28R+Y	59SR	49SR
	11	No. 042, 1.68-oz.	50PHR	50PHR	34PHY	59PHY	5R	10PHY	7R	4R+Y	15SR	15SR	29PHY	25PHR	22SR	21SR
	14½	No. 043, 1.90-oz.	3PHR	5PHR	42PHY	87PHY	7R	G	1R	2R+Y	16SR	15SR	2PHY	2R+Y	12SR	16SR
Corrosion-Resistant Uncoated Wires:																
12-14% Cr., Cold drawn	12	No. 001	M	M	SY	SY	G	G	MG	SY	71SR	71SR	89R+Y	92R+Y	48SR	46SR
	9	No. 004	M	M	SY	SY	MG	G	MG	SY	51SR	51SR	86R+Y	86R+Y	36SR	39SR
	17	No. 005	M	M	SY	SY	MG	G	MG	SY	61SR	61SR	95R+Y	93R+Y	42SR	42SR
12-14% Cr., air quenched	13	No. 002	M	M	SY	SY	MG	M	M	M	26SR	26SR	81R+Y	81R+Y	19SR	20SR
	10	No. 006	M	M	SY	SY	MG	MG	M	M	19SR	19SR	35R+Y	39R+Y	14SR	20SR
	18	No. 007	M	M	SY	SY	MG	MG	M	SY	29SR	29SR	59R+Y	53R+Y	26SR	20SR
18% Cr., 8% Ni.	15	No. 003	M	M	M	M	MG	M	M	M	MG	MG	M	M	MG	MG
	12	No. 008	M	M	M	M	M	M	M	M	MG	MG	M	M	MG	MG
	20	No. 009	M	M	SY	SY	M	M	M	M	MG	MG	M	M	MG	MG
	14	No. 015	M	M	SY	SY	M	M	M	M	-	-	M	M	-	-

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. (In cases of zinc coatings, the G and Y conditions are characteristic states of corrosion of zinc iron alloys in the coatings.) R = rust of base metal. MG = intermediate between M and G. GY predominately gray but showing indication of Y. Figures (as 5, 20, 50, 100) are percentages.

Coating Group	Average Coating, oz. per sq. ft. of Surface[a]	Wire Lot	SANDY HOOK, N.J.				BRIDGEPORT, CONN.			
			8.44 Yr.	8.92 Yr.	9.42 Yr.	9.92 Yr.	8.44 Yr.	8.92 Yr.	9.42 Yr.	9.92 Yr.
0.80 to 1.00 Avg. 0.88	1.05	No. 348.....	G	G	G	1R	G	G	20R	90R
	0.81	No. 248.....	99R	100R	100R	100R	100R	100R	100R	100R
	0.88	No. 347.....	50R	99R	100R	100R	40R	99R	100R	100R
	0.95	No. 246.....	5R	60R	65R	95R	8R	80R	85R	98R
	0.88	No. 346.....	100R	100R	100R	100R	90R	100R	100R	100R
	0.81	No. 245.....	100R	100R	100R	100R	100R	100R	100R	100R
	0.81	No. 296.....	100R	100R	100R	100R	90R	99R	99R	99R
	0.90	No. 148.....	99R	99R	100R	100R	70R	99R	100R	100R
	0.85	No. 149.....	50R	95R	95R	100R	40R	90R	90R	100R
	0.84	No. 345.....	100R	100R	100R	100R	85R	100R	100R	100R
	0.96	No. 247.....	98R	100R	100R	100R	70R	99R	99R	99R
Avg. 1.28	1.35	No. 800.....	GY	GY	GY	2R	GY	GY	GY	20R
	1.20	No. 801.....	GY	3R	10R	75R	GY	50R	60R	95R
1.60 to 1.80	2.08	No. 552.....	G	G	G	G	G	G	G	GY
	1.83	No. 451.....	G	G	G	G	G	G	G	GY
	1.75	No. 350.....	G	G	G	G	G	G	G	G
	1.69	No. 252.....	G	G	G	G	G	G	G	G
	1.65	No. 351.....	G	1R	1R	1R	G	1R	3R	3R
	1.76	No. 250.....	G	G	G	G	G	GY	15R	1R
	1.69	No. 551.....	G	G	G	G	G	GY	GY	GY
	1.50	No. 450.....	G	G	G	G	G	GY	GY	1R
	1.75	No. 352.....	G	G	G	G	G	GY	GY	1R
	1.86	No. 251.....	G	2R	2R	5R	G	GY	15R	2R
Avg. 1.76	1.86	No. 151.....	G	5R	15R	20R	G	GY	GY	1R
Heavy Avg. 2.89	2.80	No. 397.....	G	G	G	G	G	G	G	G
	2.72	No. 298.....	G	G	G	G	G	G	G	G
	3.00	No. 299.....	G	G	G	G	G	G	G	GY

In the lighter weight coating groups at Sandy Hook and at Bridgeport practically all wire had been previously reported as 100R. The only wires not reported 100R in earlier reports and their condition during the recent inspections are shown below.

Coating Group 0.60 to 0.70 at Sandy Hook
Wire No. 338...80R at 8.44 Yr., 100R at 8.92 Yr.
Wire No. 236...100R at 8.44 Yr.
Wire No. 237...100R at 8.44 Yr.

Coating Group 0.70 to 0.85 at Sandy Hook
Wire No. 442...100R at 8.44 Yr.
Wire No. 543...100R at 8.44 Yr.

Coating Group 0.60 to 0.70 at Bridgeport
Wire No. 237...90R at 8.44 Yr., 100R at 8.92 Yr.
Wire No. 135...100R at 8.44 Yr.

Coating Group 0.70 to 0.85 at Bridgeport
Wire No. 543...90R at 8.44 Yr., 100R at 8.92 Yr.

At Pittsburgh all wires had been previously reported 100R except Wire No. 298 and Wire No. 299 in the heavy coating group. Wire No. 298 was 100R at 8.47 Yr., Wire No. 299 was 35R at 8.47 Yr., 95R at 8.94 Yr., 100R at 9.44 Yr.

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[a] The zinc coating weight reported in this column is the average of all stripping test determination made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

Abbreviations and symbols used to designate appearance of states of corrosion: M = metallic; G = gray; Y = yellowed, or rust stained, but not showing actual rough rust of base metal; R = rust of base metal; PHR, PHY = rust or yellowed in pinholes; B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance; D = dark (dirt or soot) to the exclusion of a better observation; MG = intermediate M and G; GY predominately gray but showing indication of Y; RB = rust on barbs; SR = superficial rust; asterisk (*) = speckled; R+Y = indistinguishable proportions of R and Y.

Coating Group	Average Coating, oz. per sq. ft. of surface[a]	Wire[b]	State College, Pa.				Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
			7.42 yr.	7.90 yr.	8.40 yr.	8.90 yr.	8.99 yr.	9.98 yr.	8.88 yr.	9.96 yr.	8.80 yr.	9.94 yr.	9.00 yr.	9.92 yr.	8.84 yr.	9.85 yr.	8.88 yr.	9.84 yr.	8.88 yr.	9.84 yr.
0.20 to 0.30	0.30	No. C410...	100R	100R	100R	100R	100R	100R	100R	100R	GY	GY	100R	100R	G	G	5R	10R	G	G
	0.24	No. C411...	100R	100R	100R	100R	100R	100R	60R	90R	GY	GY	100R	100R	GRB	GRB	2R	5R	GRB	GRB
	0.29	No. C412...	100R	100R	100R	100R	100R	100R	15R	80R	G	GY	100R	100R	G	G	GRB	5R	G	G
0.35 to 0.45	0.37	No. C421...	60R	90R	100R	100R	100R	100R	GRB	GRB	G	GY	100R	100R	G	G	GY	Y	G	G
	0.47	No. C422...	40R	80R	95R	100R	95R	100R	GRB	10R	GY	GY	80R	80R	G	G	2R	3R	G	G
	0.31	No. C420...	100R	100R	100R	100R	100R	100R	40R	95R	G	G	100R	100R	G	GY	GY	1R	G	G
0.50 to 0.60	0.48	No. C430...	YRB	YRB	15R	30R	80R	100R	G	GRB	G	G	10R	30R	G	G	GY	2R	G	G
	0.54	No. C431...	YRB	10R	15R	20R	20R	75R	GRB	GRB	G	G	15R	20R	G	GY	2R	2R	G	G
	0.57	No. C432...	G	G	G	G	GRB	10RB	G	GRB	MG	MG	MRB	MRB	MG	MG	G	G	MG	MG
0.70 to 0.85	0.72	No. C441...	G	G	G	G	GRB	GRB	G	GRB	MG	MG	MG	MGRB	MG	MG	G	G	MG	MG
	0.78	No. C442...	G	G	G	G	GRB	GRB	G	G	MG	MG	MG	G	G	G	G	G	MG	MG
	0.74	No. C443...	GY	G	GY	GY	GRB	20R	G	G	MG	G	1R	3R	MG	MG	G	G	MG	MG
0.80 to 1.00	0.97	No. C445...	G	G	G	GY	G	G	G	G	MG	MG	G	G	G	G	G	G	MG	MG
	0.92	No. C446...	G	G	G	GY	G	G	G	G	MG	G	G	G	G	G	G	G	MG	MG
1.60 to 1.80	1.64	No. C450...	G	G	G	G	G	G	G	G	MG	MG	G	G	G	G	G	G	MG	MG
	1.50	No. C452...	G	G	G	G	MG	G	G	G	MG	MG	M	MG	G	G	G	G	MG	MG
	1.83	No. C451...	G	G	G	G	GRB	GRB	G	GRB	MG	MG	MG	MGRB	MG	MG	G	G	MG	MG
Heavy Coating	1.99	No. C494...	G	G	G	G	MG	G	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
	1.99	No. C494...	G	G	G	G	MG	G	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
	2.00	No. C447...	G	G	G	G	GRB	GRB	G	G	MG	M	M	M	G	G	G	G	MG	MG
Copper covered (3.97 oz. per sq. ft.)......		No. C025...	Gn	GnRB	GnRB	GnRB	Gn	Gn	....	Br	Br	Br	Gn	Gn	BrRB	BrRB	Br	Br	BrRB	BrRB
		No. C025...	Gn	GnRB	GnRB	GnRB	....	....	Br	....	....	....	Gn	..	....	....	....	....	....	....
Lead coated (1.83 oz. per sq. ft.)..........		No. C045...	....	....	.. .	....	10PHR	G	15PHY	90PHY	10PHY	10PHY	1R	5R	....	....	35R+Y	25R+Y	25SR	25SR
		No. C045...	....	....	.. .	....	10PHR	10R	15PHY	60PHY	....	....	....	....	20SR	25SR	35R+Y	25R+Y	....	....

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[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

[b] These barbed wire numbers do not always correspond to the unfabricated wire numbers as seen opposite:

Barbed Wire	Unfabricated Wire	Barbed Wire	Unfabricated Wire
No. C410	No. 410	No. C441	No. 442
No. C411	....	No. C442	No. 441
No. C412	No. 412	No. C443	No. 443
No. C421	....	No. C445	....
No. C422	No. 421	No. C446	....
No. C420	No. 423	No. C447	....
No. C430	No. 431	No. C450	....
No. C431	No. 430	No. C452	No. 450
No. C432	No. 432	No. C451	No. 451
		No. C494	....

TABLE III.—REPORT OF INSPECTIONS

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic; G = gray; Y = yellowed, or holes; B, Br, Gn, respectively signify corroded to a black, brown, or green appearance; D = dark (dirt or soot) to the exclusion or SR = superficial Y and superficial R, respectively; asterisk (*) = speckled appearance. Figures (as 5, 20, 50, 100) are per- stay wires. Where two values are shown separated by & (90R&Y) the first applies to the top wires of the fence and the second to

Coating Group	Average Coating, oz. per sq. ft. of surface[a]	Fence	State College, Pa.				Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.	
			7.42 yr.	7.90 yr.	8.40 yr.	8.90 yr.	8.99 yr.	9.98 yr.	8.88 yr.	9.96 yr.	8.80 yr.	9.94 yr.
0.20 to 0.30 Average 0.28	0.27	No. 512....	100R	100R	100R	100R	100R	100R	100R&G	100R&G	GY	30R
	0.30	No. 410....	100R	100R	100R	100R	100R	100R	100R&G	100R&G	10R	25R
	0.28	No. 411....	95R	100R	100R	100R	100R	100R	25R	100R&G	G	G
	0.29	No. 510....	90R	90R	95R	100R	100R	100R	30R&G	100R&G	G	G
	0.29	No. 412....	95R	99R	99R	100R	100R	100R	20R	100R&G	G	GY
	0.24	No. 511....	100R	100R	100R	100R	100R	100R	G–100R	100R&G	G	5R
0.25 to 0.35 Average 0.30	0.25	No. 315....	100R	100R	100R	100R	90R	100R	50R	100R&G	GY	20R
	0.28	No. 215....	100R	100R	100R	100R	100R	100R	80R	100R&G	GY	15R
	0.38	No. 317....	90R&Y	10R	90R	90R	90R	90R	G1R	30R	G	G
	0.32	No. 316....	100R	100R	100R	100R	90R	95R	50R	100R&G	GY	GY
	0.30	No. 216....	80R&20R	90R	95R	100R	90R	90R	10R	90R&G	G	G
0.35 to 0.45 Average 0.38	0.36	No. 520....	20R	90R	95R	100R	95R	100R	G	60R&G	G	GY
	0.35	No. 422....	55R	90R	90R	95R	90R	90R	G1R	50R	GY	GY
	0.31	No. 423....	20R	90R	90R	90R	95R	90R	G1R	50R	G	G
	0.44	No. 522....	5R	60R&Y	60R	60R	90R	90R	G	10R	G	G
	0.38	No. 521....	Y–100R	60R	80R	90R	95R	90R	G–50R	10R	G	GY
	0.47	No. 421....	60R&Y	20R–100R	20R	30R	70R	80R	G–50R	G–100R	G	GY
0.45 to 0.55 Average 0.46	0.44	No. 325....	GY	2R	5R	10R	60R	90R	G1R	G	G	GY
	0.50	No. 228....	Y	2R	10R	20R	80R	95R	G	GY	G	GY
	0.51	No. 227....	Y1R	5R	10R	20R	60R	80R	5R	10R	GY	GY
	0.38	No. 326....	G1R	10R	20R	80R	80R	90R	GY	30R	G	G
	0.47	No. 327....	Y	Y	Y	5R	40R	80R	G	G	G	G
	0.45	No. 225....	Y	Y	Y1R	1R	25R	75R	GY	GY	G	G
0.50 to 0.60 Average 0.54	0.61	No. 530....	G	G	G	G	G	15R–30R	G	G	MG	G
	0.57	No. 432....	G	G	G	G	G	G	G	G	MG	MG
	0.60	No. 531....	GY	5R	10R	15R	30R	80R	G	G–50R	MG	G
	0.48	No. 431....	Y	2R	2R	10R	60R	80R	GY	GY	G	G
	0.54	No. 430....	Y	Y	Y	5R	60R	75R	GY	GY	G	G
	0.49	No. 532....	Y	30R&Y	10R	50R	75R	80R	GY	GY	G	G
	0.49	No. 433....	Y	Y1R	Y1R	5R	70R	80R	GY	GY	G	GY
0.60 to 0.70 Average 0.64	0.69	No. 338....	G	G	G	G	G	G	G	G	MG	MG
	0.68	No. 236....	G	G	G	G	G	G	G	G	MG	MG
	0.61	No. 335....	GY	GY	GY	1R	20R	75R	G	G	MG	G
	0.63	No. 237....	GY	GY	GY	1R	10R	50R	G	G	G	G
	0.57	No. 337....	Y	Y	Y	5R	5R	40R	GY	GY	G	G
	0.67	No. 235....	Y	Y1R	5R	10R	50R	60R	GY	GY	G	G
	0.66	No. 336....	Y	Y	Y	1R	10R	50R	GY	GY	G	G
	0.60	No. 238....	Y	Y	Y	Y	5R	30R	GY	GY	G	G
0.70 to 0.85 Average 0.74	0.70	No. 540....	G	G	G	G	G	G	G	G	MG	MG
	0.72	No. 442....	G	G	G	G	G	G	G	G	MG	MG
	0.79	No. 543....	G	G	G	G	5R	25R	G	G	MG	G
	0.78	No. 441....	GY	G	G	G	G	10R	G	G	MG	MG
	0.70	No. 542....	GY	GY	GY	GY	G–10R	30R	G	G	MG	G
	0.74	No. 443....	GY	GY	GY	GY	G	30R	G	G	MG	G
	0.76	No. 541....	GY	GY	G	G	5R	20R	G	G	MG	G
	0.76	No. 440....	GY	G	G	G	G	10R	G	G	MG.	MG
0.80 to 1.00 Average 0.90	1.05	No. 348....	G	G	G	G	G	G	G	G	MG	MG
	0.81	No. 248....	G	G	G	G	MG	G	G	G	MG	MG
	0.88	No. 347....	GY	G	GY	G	G	G	G	G	MG	MG
	0.95	No. 246....	GY	G	G	G	G	G	G	G	MG	MG
	0.88	No. 346....	GY	GY	GY	GY	G	10R	G	G	MG	G
	0.81	No. 245....	GY	GY	GY	GY	G	20R	G	G	MG	MG
	0.84	No. 345....	Y	Y	Y	Y	G	5R	G	G	G	GY
	0.96	No. 247....	Y	Y	GY	Y	G	G	GY	G	G	GY
1.60 to 1.80	1.75	No. 350....	G	G	G	G	G	G	G	G	MG	MG
	1.69	No. 252....	G	G	G	G	G	MG	G	G	MG	MG
	1.75	No. 352....	G	G	G	G	G	G	G	G	MG	MG
Average 2.76	2.80	No. 397....	G	G	G	G	G	G	G	G	MG	MG
	2.72	No. 298....	G	G	G	G	G	G	G	G	MG	MG
12 to 14% Cr, cold-drawn		No. 001....	G	MG	MG	MG	M	M	SY	SY	MG	G
12 to 14% Cr, air-quenched		No. 002....	MG	MG	MG	MG	M	M	SY	SY	MG	G
18% Cr, 8% Ni		No. 003....	M	M	M	M	M	M	M	M	M	M
Copper-covered (7.42 oz. per sq. ft.)		No. 020....	Y	GnY	Gn	Gn	Gn	Gn	Br	Br	Br	Br
Lead-coated (1.48 oz. per sq. ft.)		No. 040....	80PHR	80R+Y	50R+Y	95R+Y	20R	20PHR	50PHY	80PHY	10R	[illegible]

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[a] The zinc-coating weight reported in this column is the average of all stripping test determinations made on samples

OF FARM FIELD FENCE SPECIMENS.

rust stained, but not showing actual rough rust of base metal; R = rust of base metal; PHR, PHY = rust or yellowed in pin-
of a better observation; MG = intermediate between M and G; GY = predominately gray but showing indication of Y; SY
entages. Where two values are shown separated by a dash (50R-80R) the first applies to the line wires and the second to the
the bottom wires of the fence. R+Y = indistinguishable proportions of R and Y.

Ithaca, N. Y.		Santa Cruz, Calif.		College Sta., Tex.		Davis, Calif.		Coating Group	Average Coating, oz. per sq. ft. of surface[a]	Fence
9.00 yr.	9.92 yr.	8.84 yr.	9.85 yr.	8.88 yr.	9.84 yr.	8.88 yr.	9.84 yr.			
95R&G	100R&G	G	GY	15R	30R	G	G	0.20	0.27	No. 512
70R&G	75R	GY	GY	25R	40R	G	G	to	0.30	No. 410
85R&G	85R&MG	G	GY	1R	GY	G	G	0.30	0.28	No. 411
95R&G	95R&GY	GY	GY	GY	3R	G	G	Average	0.29	No. 510
100R	100R	GY	GY	GY	3R	G	G	0.28	0.29	No. 412
95R&G	95R&GY	GY	GY	7R	20R	G	G		0.24	No. 511
100R	100R	GY	GY	12R	60R	G	G	0.25	0.25	No. 315
100R	100R	GY	GY	10R	20R	GY	1R	to	0.28	No. 215
75R	75R	G	GY	GY	GY	G	G	0.35	0.38	No. 317
85R	85R	GY	GY	GY	1R	G	G	Average	0.32	No. 316
85R	90R	GY	GY	GY	GY	G	G	0.30	0.30	No. 216
75R&G	85R&GY	GY	GY	GY	GY	G	G	0.35	0.36	No. 520
90R&G	90R&MG	GY	GY	GY	GY	G	G	to	0.35	No. 422
85R&G	90R&MG	G	GY	GY	GY	G	G	0.45	0.31	No. 423
60R&G	70R&GY	G	GY	GY	GY	G	G	Average	0.44	No. 522
70R&G	80R&GY	G	GY	GY	GY	G	G	0.38	0.38	No. 521
55R&G	75R	GY	GY	2R	GY	G	G		0.47	No. 421
55R	50R	GY	GY	GY	1R	G	G	0.45	0.44	No. 325
40R	45R	GY	GY	GY	GY	G	G	to	0.50	No. 228
35R	35R	GY	GY	GY	GY	G	G	0.55	0.51	No. 227
85R	75R	G	GY	GY	GY	G	G	Average	0.38	No. 326
15R	25R	G	GY	GY	1R	G	G	0.46	0.47	No. 327
15R	10R	GY	GY	GY	GY	G	G		0.45	No. 225
M	2R	MG	MG	G	G	MG	MG	0.50	0.61	No. 530
M	M	MG	MG	G	G	MG	MG	to	0.57	No. 432
15R&G	20R	MG	GY	G	G	MG	MG	0.60	0.60	No. 531
10R&G	30R	GY	GY	GY	GY	G	G	Average	0.48	No. 431
10R&G	50R&MG	GY	GY	GY	GY	G	G	0.54	0.54	No. 430
70R	75R	GY	GY	GY	GY	G	G		0.49	No. 532
10R	25R	GY	GY	GY	GY	G	G		0.49	No. 433
M	M	MG	MG	G	G	MG	MG	0.60	0.69	No. 338
M	M	MG	MG	G	G	MG	MG	to	0.68	No. 236
G	5R	G	MG	G	G	MG	MG	0.70	0.61	No. 335
G	2R	G	G	G	G	MG	MG	Average	0.63	No. 237
1R	5R	GY	GY	GY	2R	G	G	0.64	0.57	No. 337
25R	30R	GY	GY	2R	2R	G	G		0.67	No. 235
5R	5R	10Y	GY	1R	4R	G	G		0.66	No. 336
1R	5R	GY	GY	1R	3R	G	G		0.60	No. 238
M	M	MG	MG	G	G	MG	MG	0.70	0.70	No. 540
M	M	MG	MG	G	G	MG	MG	to	0.72	No. 442
MG	MG	MG	MG	G	G	MG	MG	0.85	0.79	No. 543
MG	MG	MG	MG	G	G	MG	MG	Average	0.78	No. 441
G	3R	MG	G	G	G	MG	MG	0.74	0.70	No. 542
G	G	MG	MG	G	G	MG	MG		0.74	No. 443
MG	1R	MG	MG	G	G	MG	MG		0.76	No. 541
MG	MG	MG	MG	G	G	MG	MG		0.76	No. 440
M	M	MG	MG	G	G	MG	MG	0.80	1.05	No. 348
M	M	MG	MG	G	G	MG	MG	to	0.81	No. 248
MG	MG	G	G	G	G	MG	MG	1.00	0.88	No. 347
MG	G	G	G	G	G	MG	MG	Average	0.95	No. 246
G	G	MG	MG	G	G	MG	MG	0.90	0.88	No. 346
G	GY	G	MG	G	G	MG	MG		0.81	No. 245
G	1R	G	G	GY	2R	G	G		0.84	No. 345
G	GY	G	GY	GY	2R	G	G		0.96	No. 247
M	M	MG	MG	G	G	MG	MG	1.60	1.75	No. 350
M	M	MG	MG	G	G	MG	MG	to	1.69	No. 252
M	M	G	G	G	G	G	G	1.80	1.75	No. 352
M	M	MG	MG	G	G	MG	MG	Average	2.80	No. 397
M	M	MG	MG	G	G	MG	MG	2.76	2.72	No. 298
M•	MG•	40SR	50SY	10R	5R	75SR	90SR	12 to 14% Cr, cold-drawn		No. 001
M	M	20SR	25SR	50SY	1SR	30SR	10SR-35SR	12 to 14% Cr, air-quenched		No. 002
M	M	M5G	MG	M5SY•	5SY	M5G	MG	18% Cr, 8% Ni		No. 003
B	Gn	Br	Br	Br	Br	Br	Br	Copper-covered (7.42 oz. per sq. ft.)		No. 020
2R	5R	30SR	40SR	30PHS	5R	30SR	50SR	Lead-coated (1.48 oz. per sq. ft.)		No. 040

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unfabricated wire, fence or barbed wire of the specific wire lot number.

TABLE IV.—REPORT OF INSPECTIONS OF CHAIN LINK SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic; G = gray; Y = yellowed, or rust stained, but not showing actual rough rust of base metal; R = rust of base metal; PHR, PHY = rust or yellowed in pinholes; B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance; D = dark (dirt or soot) to the exclusion of a better observation; MG = intermediate between M and G; GY predominately gray but showing indication of Y; Asterisk (*) = sample disappearance—probably stolen; R + Y = indistinguishable proportions of R and Y; SR = superficial rust.

All 200 series wires are 9 gage. Lead-coated wire, 9 gage. Corrosion-resistant steel, 14 gage.

All 100 series wires are 6 gage. Copper-covered wire 0.128D.

Zinc Coating, oz. per sq. ft. of surface, by stripping test	Fence	Pittsburgh, Pa.			Sandy Hook, N. J.				Bridgeport, Conn.			
		8.47 yr.	8.94 yr.	9.44 yr.	8.94 yr.	8.92 yr.	9.42 yr.	9.92 yr.	8.44 yr.	8.92 yr.	9.42 yr.	9.92 yr.
0.29	No. E280[a]	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
0.52	No. E281	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
0.37	No. E282	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
1.22	No. E185	100R	100R	100R	10R	10R	20R	50R	GY	2R	20R	30R
1.13	No. E285	100R	100R	100R	10R	20R	60R	75R	GY	2R	20R	20R
2.92	No. E290	100R	100R	100R	G	G	G	G	G	G	G	G
2.09	No. E291	100R	100R	100R	G	G	G	G	G	GY	GY	GY
1.60	No. H280[a]	100R	100R	100R	50R	50R	50R	60R	GY	50R	40R	60R
1.83	No. H185	108R	100R	100R	10R	10R	10R	20R	GY	2R	2R	20R
1.60	No. H186	105R	100R	100R	GY	1R	Y	5R	GY	GY	Y	5R
1.61	No. H187	100R	100R	100R	GY	5R	2R	15R	GY	GY	GY	5R
1.55	No. H188	100R	100R	100R	5R	35R	40R	60R	GY	30R	50R	60R
1.36	No. H285	100R	100R	100R	5R	30R	20R	40R	GY	GY	Y	40R
1.97	No. H286	100R	100R	100R	5R	20R	20R	25R	GY	GY	Y	20R
1.46	No. H287	100R	100R	100R	25R	30R	50R	50R	GY	40R	50R	60R
1.74	No. H288	100R	100R	100R	GY	GY	Y	GY	GY	GY	Y	GY
1.40	No. H289[b]	100R	100R	100R	G	G	G	G	GY	G	G	G
4.21	No. H290	40R	50R	50R	G	G	G	GY	G	GY	G	GY
Lead-Coated L050 (1.75 oz. per sq. ft.)		35R	30R	35R	50R+Y	50R+Y	60R+Y	60R+Y	10PHR	5PHR	Y	Y
Copper-covered K030 (8.52 oz. per sq. ft.)		*	*	*	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn
Corrosion-resistant steel J011		D	D	D	GY	MG	MG	MG	MG	M	MG	MG

Zinc Coating, oz. per sq. ft. of surface, by stripping test	Fence	State College, Pa.				Lafayette, Ind.		Ithaca, N. Y.		Santa Cruz, Calif.		Davis, Calif.	
		7.42 yr.	7.90 yr.	8.40 yr.	8.90 yr.	8.99 yr.	9.98 yr.	9.00 yr.	9.92 yr.	8.84 yr.	9.85 yr.	8.88 yr.	9.84 yr.
0.29	No. E280[a]	100R	100R	100R	100R	100R	100R	100R	100R	GY	GY	G	10R
0.52	No. E281	GY	5R	20R	30R	80R	90R	60R	75R	GY	GY	G	G
0.37	No. E282	50R	99R	100R	100R	100R	100R	90R	95R	GY	GY	G	G
1.22	No. E185	GY	GY	GY	GY	G	G	MG	G	MG	MG	MG	MG
1.13	No. E285	GY	GY	GY	GY	G	G	MG	G	MG	MG	MG	MG
2.92	No. E290	G	G	G	G	G	MG	M	G	MG	MG	MG	MG
2.09	No. E291	G	G	G	G	G	MG	M	M	MG	MG	MG	MG
1.60	No. H280[a]	G	G	G	G	G	G	G	G	G	MG	G	MG
1.83	No. H185	G	G	G	GY	G	G	G	G	MG	MG	MG	MG
1.60	No. H186	G	G	G	G	G	G	G	G	MG	MG	MG	MG
1.61	No. H187	G	G	G	G	G	G	G	G	MG	MG	G	MG
1.55	No. H188	G	G	G	G	G	MG	G	G	MG	MG	G	MG
1.36	No. H285	G	G	G	GY	G	G	G	G	G	G	MG	MG
1.97	No. H286	G	G	G	GY	G	G	G	G	MG	MG	MG	MG
1.46	No. H287	G	G	G	GY	G	G	G	G	MG	MG	MG	MG
1.74	No. H288	G	G	G	G	G	G	G	G	MG	MG	MG	MG
1.40	No. H289[b]	G	G	G	G	G	G	G	M	MG	MG	MG	MG
4.21	No. H290	G	G	G	GY	MG	MG	M	M	MG	MG	MG	MG
Lead-Coated L050 (1.75 oz. per sq ft.)		70R+Y	70R+Y	70R+Y	90R+Y	20R	40PHR	3R	5R	25SR	20SR	35SR	35SR
Copper-covered K030 (8.52 oz. per sq. ft.)		Gn	Gn	Gn	Gn	Gn	Gn	Br	Br	Br	Br	Br	Br
Corrosion-resistant steel J011		M	M	M	M	M	M	M	M	MG	MG	MG	MG

[a] E numbers, coating applied before weaving H numbers, coating applied after weaving.

[b] While this sample has an H number, its appearance indicates that it was galvanized before weaving.

TABLE V.—REPORT OF INSPECTIONS OF WIRE STRAND SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray; Y = yellowed, or rust-stained, but not showing actual rough rust of base metal; R = rust of base metal; PHR, PHY = rusted or yellowed in pinholes; B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance; D = dark (dirt or soot) to the exclusion of a better observation; MG = intermediate between M and G; GY predominately gray but showing indication of Y; R+Y = indistinguishable proportions of R and Y.

Zinc Coating, oz. per sq. ft. of surface	Strand	Pittsburgh, Pa.			Sandy Hook, N. J.				Bridgeport, Conn.				State College, Pa.				Lafayette, Ind.	
		8.47 yr.	8.94 yr.	9.44 yr.	8.44 yr.	8.92 yr.	9.42 yr.	9.92 yr.	8.44 yr.	8.92 yr.	9.42 yr.	9.92 yr.	8.29 yr.	8.77 yr.	9.27 yr.	9.77 yr.	8.99 yr.	9.98 yr.
0.69	No. D345....	100R	90R	100R	G	1R	5R	10R	G	2R	10R	10R	G	G	G	G	M	MG
0.88	No. D346....	100R	80R	100R	GY	GY	GY	1R	GY	GY	GY	GY	GY	GY	G	G	G	G
0.87	No. D347....	100R	100R	100R	GY	1R	GY	2R	GY	GY	GY	GY	GY	G	GY	G	G	G
0.93	No. D348....	100R	100R	100R	G	2R	5R	2R	G	2R	2R	5R	G	G	G	G	M	MG
1.10	No. D360....	100R	100R	100R	G	G	G	G	G	G	G	GY	G	G	G	G	M	MG
1.17	No. D361....	100R	98R	100R	GY	GY	GY	GY	G	GY	GY	GY	GY	G	G	G	G	G
1.03	No. D362....	100R	98R	100R	GY	GY	GY	GY	GY	GY	Y	Y	GY	GY	GY	GY	G	G
1.51	No. D365....	60R	70R	90R	GY	GY	GY	GY	GY	GY	GY	GY	GY	GY	G	G	G	G
1.39	No. D366....	50R	50R	50R	G	G	G	G	G	G	G	G	G	GY	G	G	M	MG
1.32	No. D367....	100R	20R	80R	GY	GY	GY	GY	GY	GY	Y	GY	GY	GY	Y	GY	G	G
1.71	No. D371....	50R	60R	50R	G	G	G	G	GY	GY	G	GY	G	G	G	G	M	MG
2.47	No. D375....	5R	25R	30R	G	G	G	G	G	G	G	G	G	G	G	G	M	MG
Lead coating (1.96 oz. per sq. ft.).......	No. D045....	20R	30R	30R	90R+Y	95R+Y	90R+Y	90R+Y	20PHR	50R+Y	70R+Y	70R+Y	90R+Y	70R+Y	70R+Y	95R+Y	25R	35PHR

Zinc Coating, oz. per sq. ft. of surface	Strand	Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
		8.88 yr.	9.96 yr.	8.80 yr.	9.94 yr.	9.00 yr.	9.92 yr.	8.84 yr.	9.85 yr.	8.88 yr.	9.84 yr.	8.88 yr.	9.84 yr.
0.69	No. D345....	G	G	MG	MG	M	M	MG	G	G	G	G	G
0.88	No. D346....	G	G	MG	MG	MG	MG	MG	G	G	G	MG	MG
0.87	No. D347....	G	G	MG	MG	MG	MG	G	G	G	G	G	G
0.93	No. D348....	G	G	G	MG	M	MG	G	G	G	G	MG	G
1.10	No. D360....	G	G	MG	MG	M	M	G	G	G	G	MG	G
1.17	No. D361....	G	G	MG	MG	MG	MG	G	G	G	G	MG	G
1.03	No. D362....	G	G	G	MG	G	G	MG	MG	G	G	MG	G
1.51	No. D365....	G	G	MG	MG	MG	G	G	G	G	G	G	G
1.39	No. D366....	G	G	MG	MG	M	M	MG	G	G	G	MG	G
1.32	No. D367....	G	G	MG	MG	G	G	MG	MG	G	G	MG	G
1.71	No. D371....	G	G	MG	MG	M	M	MG	G	G	G	MG	G
2.47	No. D375....	G	G	MG	MG	M	M	MG	G	G	G	MG	G
Lead coating (1.96 oz. per sq. ft.).......	No. D045....	70PHY	95PHY	10R	30Y	2R	3R	45SR	45SR	80R+Y	75PHY4R	50SR3R	45SR

TABLE VI.—BREAKING LOADS AND ELONGATION OF UNFABRICATED WIRE SEPCIMENS.

Wire Lot	Exposure, yr.	First Rust, yr.	Breaking Load, lb.	Elongation in 10 in., per cent	Wire Lot	Exposure, yr.	First Rust, yr.	Breaking Load, lb.	Elongation in 10 in., per cent
TESTS ON SPECIMENS EXPOSED AT BRUNOT ISLAND (PITTSBURGH, PA.)									
No. 552	8.47	4.90	90	0.4	No. 246	9.44	2.90	830	10.0
No. 552	9.44	4.90	70	0.2	No. 247	9.44	2.90	1295	3.1
No. 450	8.47	2.95	350	0.3	No. 248	9.44	2.10	995	0.5
No. 450	9.44	2.95	285	0.4	No. 296	9.44	2.50	1110	2.2
No. 451	8.47	4.50	270	1.8	No. 250	8.47	3.90	1125	7.8
No. 451	9.44	4.50	255	0.8	No. 251	8.47	3.90	1525	0.8
No. 315	9.44	0.45	345	4.0	No. 252	8.47	4.50	900	7.7
No. 316	9.44	1.20	480	0.6	No. 298	8.47	7.70	1440	6.4
No. 317	9.44	1.45	325	7.4	No. 298	9.44	7.70	1375	4.0
No. 325	9.44	1.75	340	10.5	No. 299	8.47	7.00	1745	0.5
No. 326	9.44	1.40	180	2.7	No. 299	9.44	7.00	1620	1.3
No. 327	9.44	1.80	525	0.9	No. 151	8.47	3.90	2690	0.4
No. 329	9.44	1.85	325	8.9	No. 800	8.47	3.90	2240	4.3
No. 335	9.44	1.60	480	0.6	No. 801	8.47	3.50	1115	2.7
No. 336	9.44	2.75	315	7.7	No. 001	8.47	....	1290	2.6
No. 337	9.44	2.50	395	3.8	No. 002	8.47	....	1290	3.3
No. 338	9.44	2.00	510	0.6	No. 003	8.47	....	1240	0.3
No. 345	9.44	2.70	405	4.9	No. 004	8.47	....	2370	3.5
No. 346	9.44	2.55	535	1.0	No. 005	8.47	....	315	1.5
No. 347	9.44	2.65	460	2.1	No. 006	8.47	....	2510	6.8
No. 348	9.44	3.20	525	0.8	No. 007	8.47	....	265	1.1
No. 350	8.47	5.10	475	6.8	No. 008	8.47	....	2545	0.9
No. 351	8.47	2.90	665	5.8	No. 009	8.47	....	290	....
No. 352	8.47	3.90	905	0.6	No. 015	8.47	....	1270	0.5
No. 397	8.47	7.50	800	2.2	No. 021	8.47	....	1660	1.0
No. 397	9.44	7.50	725	0.9	No. 022	8.47	....	2330	1.1
No. 245	9.44	2.40	1050	2.0					
TESTS ON SPECIMENS EXPOSED AT STATE COLLEGE, PA.									
No. 501	9.27	0	335	0.4	No. 406	9.27	0	385	11.1
No. 506	9.27	0	325	0.5	No. 421	9.27	5.80	460	14.1
No. 507	9.27	0	365	0.5	No. 422	9.27	6.80	515	14.1
No. 520	9.27	6.30	290	14.9	No. 423	9.27	5.80	395	15.4
No. 521	9.27	6.80	295	14.0	No. 315	9.27	3.70	870	9.1
No. 522	9.27	7.50	520	0.5	No. 316	9.27	5.70	1090	4.4
No. 400	9.27	0	350	11.3	No. 317	9.27	6.80	815	10.9
No. 401	9.27	0	360	9.3	No. 282	9.27	6.70	1310	11.5
No. 405	9.27	0	380	11.3					
TESTS ON SPECIMENS EXPOSED AT BRIDGEPORT, CONN.									
No. 501	8.44	0	280	0.4	No. 423	8.44	2.50	350	13.7
No. 506	8.44	0	285	0.4	No. 430	8.44	4.80	715	1.5
No. 507	8.44	0	305	0.2	No. 431	8.44	3.50	750	2.5
No. 510	8.44	2.50	360	0.2	No. 432	8.44	5.40	485	5.9
No. 511	8.44	1.80	185	10.6	No. 440	8.44	4.80	525	10.7
No. 512	8.44	1.60	210	8.3	No. 440	9.42	4.80	470	10.6
No. 520	8.44	2.50	220	12.2	No. 441	8.44	5.90	475	13.9
No. 520	9.42	2.50	235	10.9	No. 441	9.42	5.90	425	12.2
No. 521	8.44	3.40	200	10.4	No. 442	8.44	6.30	410	9.4
No. 521	9.42	3.40	210	10.2	No. 442	9.42	6.30	375	11.2
No. 522	8.44	3.40	340	0.2	No. 443	8.44	5.80	660	1.6
No. 522	9.42	3.40	330	0.3	No. 443	9.42	5.80	610	1.4
No. 530	8.44	5.40	235	7.1	No. 444	8.44	5.60	555	12.3
No. 530	9.42	5.40	225	5.5	No. 444	9.42	5.60	545	11.9
No. 531	8.44	4.70	375	0.4	No. 495	8.44	5.90	660	1.9
No. 531	9.42	4.70	330	0.3	No. 495	9.42	5.90	610	1.0
No. 540	8.44	6.20	250	4.6	No. 325	8.44	3.90	660	18.0
No. 540	9.42	6.20	245	4.4	No. 326	8.44	2.90	600	14.8
No. 541	8.44	4.90	325	11.7	No. 327	8.44	3.80	1000	4.3
No. 541	9.42	4.90	300	10.9	No. 329	8.44	3.80	660	13.5
No. 542	8.44	5.50	390	0.3	No. 335	8.44	5.40	910	4.2
No. 542	9.42	5.50	355	0.4	No. 335	9.42	5.40	900	3.8
No. 543	8.44	5.90	260	8.8	No. 336	8.44	5.40	700	15.0
No. 543	9.42	5.90	250	6.5	No. 336	9.42	5.40	665	13.9
No. 400	8.44	0	345	11.9	No. 337	8.44	5.40	770	12.8
No. 401	8.44	0	315	11.1	No. 337	9.42	5.40	720	11.6
No. 405	8.44	0	360	12.2	No. 338	8.44	6.70	945	1.1
No. 406	8.44	0	370	13.9	No. 338	9.42	6.70	890	1.0
No. 421	8.44	2.70	415	13.9	No. 345	8.44	5.70	780	13.2
No. 422	8.44	2.70	445	13.0	No. 345	9.42	5.70	845	12.3

Continued on p. 136

TABLE VI —*Continued.*

Wire Lot	Exposure, yr.	First Rust, yr.	Breaking Load, lb.	Elongation in 10 in., per cent	Wire Lot	Exposure, yr.	First Rust, yr.	Breaking Load, lb.	Elongation in 10 in., per cent
TESTS ON SPECIMENS EXPOSED AT BRIDGEPORT, CONN.—*Continued.*									
No. 346......	8.44	6.90	995	4.7	No. 239......	8.44	4.40	1065	15.1
No. 346......	9.42	6.90	935	5.1	No. 282......	8.44	2.80	1240	10.2
No. 347......	8.44	6.90	785	6.9	No. 245......	8.44	5.90	1530	5.3
No 347......	9.42	6.90	790	5.7	No. 246......	8.44	6 90	1190	12.1
No. 348......	8.44	....	965	0.9	No. 247......	8.44	5.90	1705	7.1
No. 348......	9.42	....	950	0.9	No. 248......	8.44	6.80	1379	1.6
No. 225......	8.44	3.90	1550	4.8	No. 296......	8.44	6.90	1560	5.9
No. 226......	8.44	3.90	1090	13.3	No. 125......	9.42	3.70	1755	15.7
No. 227......	8.44	4.40	1480	5.7	No. 127......	9.42	4.00	2380	5.2
No. 228......	8.44	3.80	1015	14.4	No. 135......	8.44	4.90	2550	5.0
No. 235......	8.44	5.70	1670	5.8	No. 138......	8.44	4.90	1880	15.2
No. 236......	8.44	6.80	1345	2.6	No. 139......	8.44	4.90	1855	11.9
No. 237......	8.44	4.90	1465	3.1	No. 148......	8 44	6.90	2440	6.1
No. 238......	8.44	4.90	1550	6.6	No. 149......	8.44	6.90	1825	14.4
TESTS ON SPECIMENS EXPOSED AT SANDY HOOK, N. J.									
No. 510......	8.44	1.75	255	0.2	No. 338......	9.42	7.90	925	1.0
No. 511......	8.44	1.75	115	2.4	No. 345......	8.44	5.90	770	10.3
No. 512......	8.44	1.55	145	2.5	No. 345......	9.42	5.90	690	10.8
No. 520......	8.44	2.70	165	4.7	No. 346......	8.44	6.50	925	4.2
No. 521......	8.44	2.90	145	3.7	No. 346......	9.42	6.50	875	4.2
No. 522......	8.44	2.90	260	0.2	No. 347......	8 44	7.80	830	5.0
No. 530......	8.44	2.90	220	6.3	No. 347......	9.42	7.80	745	6.5
No. 530......	9.42	2 90	200	4.1	No. 348......	8 44	....	960	1.2
No. 531......	8.44	4.70	335	0.3	No. 348......	9 42	....	945	0 9
No. 531......	9.42	4.70	275	0.5	No. 225......	8.44	3.90	1435	3.9
No 540......	8.44	5.90	245	5 0	No 225......	9 42	3.90	1390	3.7
No. 540......	9.42	5.90	235	3.6	No. 226......	8.44	3.90	1035	15.1
No. 541......	8.44	5.40	260	7.5	No 226......	9.42	3.90	1015	14.8
No. 541......	9.42	5.40	230	5.9	No. 227......	8 44	4.40	1405	5.1
No 542......	8.44	5.50	330	0.2	No. 227......	9 42	4.40	1360	4.7
No. 542......	9.42	5.50	335	0 2	No. 228......	8.44	2.90	960	13.5
No. 543......	8.44	5.90	250	6.3	No. 228......	9.42	2.90	955	14.0
No. 543......	9.42	5.90	245	3.1	No. 235......	8 44	5.50	1210	11.6
No. 430......	8.44	4.40	620	0.8	No. 235......	9.42	5.50	1190	12.1
No. 430......	8.44	4.40	635	1.0	No 236......	8.44	7.70	1355	1.9
No. 430......	9.42	4.40	580	0.7	No. 236......	9 42	7.70	1360	2.6
No. 431......	8.44	3.70	655	1.2	No. 237......	8 44	4.40	1425	4.0
No. 431......	9.42	3.70	605	1.1	No. 237......	9.42	4.40	1340	2.6
No. 432......	8.44	6.50	455	5.1	No. 238......	8 44	4.80	1435	5.8
No. 432......	9.42	6.50	430	3.2	No 238......	9.42	4 80	1405	4.1
No. 440......	8.44	4.70	435	8.9	No 239......	8.44	5.50	1035	17.0
No. 440......	9.42	4.70	425	7 7	No. 239......	9.42	5.50	1025	16.9
No. 441......	8.44	6.50	460	12.9	No. 245......	8.44	5.90	1460	5.2
No. 441......	9.42	6.50	425	9.5	No. 245......	9.42	5.90	1445	6 5
No. 442......	8.44	7.40	380	11.5	No. 246......	8.44	7.90	1200	9.3
No. 442......	9.42	7.40	425	10.2	No. 246......	9.42	7.90	1160	10.4
No. 443......	8.44	5.90	615	1.3	No 247......	8 44	6.50	1500	5.8
No. 443......	9.42	5.90	595	0.9	No 247......	9.42	6.50	1395	8 7
No. 444......	8.44	5.70	465	10.5	No 248......	8.44	7.40	1375	1.8
No. 444......	9.42	5.70	470	7.2	No. 248......	9 42	7.40	1340	1.3
No. 495......	8.44	5.90	620	1.2	No. 296......	8.44	5 90	1460	5.1
No. 495......	9.42	5.90	615	1.0	No. 296......	9.42	5 90	1435	4.5
No. 325......	8.44	3.80	635	15.1	No 125......	9.42	3.45	1655	14.0
No. 325......	9.42	3.80	630	14.4	No 127......	9.42	3.90	2305	5.2
No. 326......	8.44	3.85	580	13.7	No 135......	8.44	5.90	2460	4.9
No. 326......	9.42	3.85	545	11.8	No 135......	9.42	5.90	2390	4.5
No. 327......	8.44	3.80	900	2 5	No. 138......	8.44	5.50	1800	15.0
No. 327......	9.42	3.80	890	2 0	No 138......	9.42	5.50	1815	14.4
No. 329......	8.44	3.80	600	13 1	No. 139......	8 44	4.80	1745	11.3
No. 329......	9.42	3.80	580	11.5	No 139......	9 42	4.80	1755	13.1
No. 335......	8.44	4.90	855	3.4	No 148......	8 44	6.90	2480	6.4
No. 335......	9.42	4.90	840	1.9	No 148......	9.42	6.90	2405	5.1
No 336......	8.44	4.80	650	13.6	No. 149......	8 44	6.80	1870	15.2
No. 336......	9.42	4.80	600	11.8	No. 149......	9.42	6.80	1840	13.0
No. 337......	8.44	4.80	690	9.9	No. 800......	9.42	....	2535	7.4
No. 337......	9.42	4 80	665	8.2	No. 801......	9.42	8.90	1455	5.4
No. 338......	8.44	7.90	965	1.2					

PLATE II
PROCEEDINGS, AM. SOC. TESTING MATS.
VOL. 47
REPORT OF COMMITTEE A-5:
WIRE INSPECTION REPORT

REFERENCE INFORMATION
ON UNFABRICATED WIRE, BARBED WIRE AND FENCE IN THE WIRE INSPECTION REPORT.

The Wire Test Committee responsible for planning and arranging the exposure tests of specimens covered in this report recommends that the following statement be made in all reports relating to the samples of wire and wire products under exposure tests:

> "It is emphasized that the weight of coating ranges shown under each weight group have been arbitrarily chosen and purposely held to close limitations by the Committee and do not necessarily individually represent any manufacturers' commercial product. On the other hand, the entire range of coatings covered by all weight groups as a whole are designed to include a sufficient range of coating to furnish the useful corrosion resistance data desired."

SYSTEM OF NUMBERING SPECIMENS

Zinc coated and uncoated steel specimens (all have 3-digit numbers)......................(c)

When the first digit is......	5	4	3	2	1
the respective gages are:	14.5	12.5	11	9	6

The second and third digits indicate weight of zinc coating as follows:

	oz. per sq.ft.		oz. per sq.ft.
00–04 (LC)*	uncoated	50–59 (CB)	1.6 to 1.8
05–09 (CB)*	uncoated	60–64 (CB)	1.0 to 1.2
10–14 (CB)	0.20 to 0.30	65–69 (CB)	1.3 to 1.5
15–19 (CB)	0.25 to 0.35	70–74 (CB)	1.6 to 1.8
20–24 (CB)	0.35 to 0.45	75–79 (CB)	2.4 to 2.6
25–29 (CB)	0.45 to 0.55	80–84 (CB)	less than 0.50
30–34 (CB)	0.50 to 0.60	85–89 (CB)	1.2 to 1.5
35–39 (CB)	0.60 to 0.70	90–94 (CB)	2.4 to 3.0
40–44 (CB)	0.70 to 0.85	97–99 (CB)	extra heavy
		95–96 (CB)	chromated after coating
45–49 (CB)	0.80 to 1.00 on farm fence and unfabricated wire. 0.85 to 1.00 on barbed wire. 0.80 to 0.95 on wire strand.		

Specimens of Special Materials:
000–015: uncoated corrosion-resistant steel
020–030: copper-covered steel.
040–050: lead-coated steel.

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* LC = low copper content wire; CB = copper bearing wire

Reference Characters

(a)......Wire lot numbers identify wire from a coil or coils of wire supposedly alike.

Wire Lots of Zinc-Coated or Bare Mild Steel	Usually Appear in the Test as shown by ✓				
	Unfab. + Wire	Int. of + Fence	T and B + of Fence	T and B of Fence	Barbed Wire
100 series	✓ (S)	....	....	....	....
200 series	✓ (S)	✓ (S)	✓ (S)	✓ (300)	....
300 series	✓ (S)	✓ (S)	....	✓ (500)	....
400 series	✓ (S)	✓ (S)	....	....	✓ (400)
(s)...... 500 series	✓ (S)	✓ (S)	....	....	....

(S) The specimen numbers of the unfabricated wire or fence are the same as the wire lot numbers.

(300), (400), (500): Specimens of fence numbered in the series 300 have top and bottom wires of the 200 series wire lots; fences numbered in the 500 series have top and bottom wires of the 300 series wire lots. Specimens of barbed wire (numbered in the 400 series) have wire lots of the 400 series but not necessarily of the same identical wire lot numbers.

Par. A. The zinc-coated wire lots table below shows:

(1) All the individual numbered wire lots and the various utilizations of each wire lot in the test.

(2) The results of weight of coating tests on samples cut from each source (unfabricated wire, fence interiors, fence top and bottom wires, and barbed wires).

(b)...... (3) The average coating for each wire lot which is the average of all the figures in the third and following columns in each row.

These average coating figures are the ones entered in other tables of this report. For example, wherever unfabricated wire sample No. 317 or farm fence sample No. 317 are listed, their indicated weights of coating are 0.38 oz. per sq.ft., and not 0.46 and 0.34, respectively.

These wire lot and coating reference data provide an indication of the uniformity of coating on any wire lot.

Par. B. The makeup of fences table below shows:

(1) The lot number of the wire used in the top and bottom line wires of each fence. (The lot number of the wire used in the interior line and stay wires of any fence is the same as the fence number.)

(2) The type of fence, that is, the combination of gages used in its fabrication as explained below:

(d)......				
Type of fence	4	3	2	1
Gage of interior line and stay wires	14.5	12.5	11	9
Gage of top and bottom line wires	11	10	9	9

+ ......Abbreviations: Unfab., unfabricated wire; Int., interior line and stay wire; T and B, top and bottom line wires.

ZINC-COATED WIRE LOTS: THEIR COATING WEIGHTS AND TEST SPECIMENS IN WHICH THEY ARE USED
(See Par. A and reference characters above for explanations) (Coating weights, oz. per sq.ft.)

Wire Lot (a) (Fence)	(b) Average Coating	Coating Weight Tests on Samples Cut From Unfab. Wire(s)	Coating Weight Tests on Samples Cut From Fence Int.(s)
No. 510(c)	0.29	0.29	0.29
No. 511	0.24	0.24	0.23
No. 512	0.27	0.27	0.27
No. 520	0.36	0.34	0.38
No. 521	0.38	0.40	0.36
No. 522	0.44	0.44	0.44
No. 530	0.61	0.59	0.62
No. 531	0.60	0.58	0.61
No. 532	0.49	....	0.49
No. 540	0.70	0.72	0.68
No. 541	0.76	0.80	0.72
No. 542	0.70	0.69	0.71
No. 543	0.79	0.83	0.74
No. 551	1.69	1.69	....
No. 552	2.08	2.08	....

Wire Lot (a) (Fence)	(b) Average Coating	Unfab. Wire(s)	Fence Int.(s)	Barbed Wire Coating	Barbed Wire No.
No. 410(c)	0.30	0.25	0.38	0.27	(410)
No. 411	0.28	0.28	0.28	....	....
No. 412	0.29	0.30	0.30	0.27	(412)
No. 421	0.47	0.49	0.49	0.44	(422)
No. 422	0.35	0.38	0.31	....	....
No. 423	0.31	0.35	0.31	0.28	(420)
No. 430	0.54	0.56	0.52	0.53	(431)
No. 431	0.48	0.49	0.48	0.46	(430)
No. 432	0.57	0.58	0.60	0.53	(432)
No. 433	0.49	....	0.49	....	....
No. 440	0.76	0.72	0.79	....	....
No. 441	0.78	0.77	0.76	0.82	(442)
No. 442	0.72	0.65	0.86	0.64	(441)
No. 443	0.74	0.74	0.78	0.70	(443)
No. 444	0.87	0.87	....	....	....
No. 450	1.50	1.48	....	1.52	(452)
No. 451	1.83	1.71	....	1.95	(451)
No. 495 Cr	Made from unfab. wire 443.				

	Unfab. Wire		Unfab. Wire
No. 115(c)	0.25	No. 139	0.64
No. 117	0.27	No. 148	0.90
No. 125	0.41	No. 149	0.85
No. 127	0.54	No. 151	1.86
No. 135	0.74	No. 800(6 ga.)	1.35
No. 138	0.67	No. 801(9 ga.)	1.20

Wire Lot (a) (Fence)	(b) Average Coating	Coating Weight Tests on Samples Cut From Unfab. Wire(s)	Fence Int.(s)	T and B of Fence Top	T and B of Fence Bottom	No.
No. 315(c)	0.25	0.25	0.25	0.25	0.26	(512)
No. 316	0.32	0.32	0.31	0.32	0.33	(510)
No. 317	0.38	0.46	0.34	0.37	0.36	(511)
No. 325	0.44	0.50	0.52	0.37	0.39	(520)
No. 326	0.38	0.40	0.35	....	....	....
No. 327	0.47	0.48	0.44	0.48	0.48	(522)
No. 329	0.54	0.56	....	0.45	0.61	(521)
No. 335	0.61	0.61	0.63	0.59	0.60	(531)
No. 336	0.66	0.73	0.60	....	....	....
No. 337	0.57	0.66	0.59	0.50	0.52	(532)
No. 338	0.69	0.67	0.76	0.67	0.65	(530)
No. 345	0.84	0.83	0.97	0.76	0.79	(541)
No. 346	0.88	0.84	0.84	0.96	0.88	(542)
No. 347	0.88	0.96	1.00	0.78	0.79	(543)
No. 348	1.05	1.04	1.07	1.05	1.06	(540)
No. 350	1.75	1.73	1.76	....	....	....
No. 351	1.65	1.65	....	....	....	....
No. 352	1.75	1.82	1.68	....	....	....
No. 397	2.80	2.80	2.79	....	....	....

Wire Lot (a) (Fence)	(b) Average Coating	Unfab. Wire(s)	Fence(s) T-B Top	Fence(s) T-B Bottom	T and B of Fence Top	T and B of Fence Bottom	No.
No. 215(c)	0.28	0.28	0.25	0.25	0.33	0.29	(315)
No. 216	0.28	0.31	0.30	0.31	0.27	0.29	(316)
No. 217	0.36	0.29	....	....	0.40	0.39	(317)
No. 225	0.45	0.45	0.49	0.44	0.43	0.47	(327)
No. 226	0.45	0.46	....	....	0.45	0.45	(326)
No. 227	0.51	0.59	0.48	0.49	....	....	
No. 228	0.50	0.49	0.48	0.48	0.50	0.53	(325)
No. 235	0.67	0.82	0.60	0.62	0.68	0.64	(337)
No. 236	0.68	0.66	0.68	0.73	0.69	0.65	(338)
No. 237	0.63	0.72	0.67	0.55	0.58	0.61	(335)
No. 238	0.60	0.60	0.59	0.61	0.59	0.61	(336)
No. 239	0.65	0.65	....	....	....	....	....
No. 245	0.81	0.80	0.83	0.78	0.81	0.81	(346)
No. 246	0.95	0.97	0.94	0.98	0.89	0.95	(347)
No. 247	0.96	0.94	0.95	1.00	1.01	0.91	(345)
No. 248	0.81	0.66	0.92	0.68	0.91	0.89	(348)
No. 250	1.76	1.76	....	....	....	....	....
No. 251	1.86	1.68	....	....	1.99	1.91	(352)
No. 252	1.69	1.67	1.85	1.57	....	....	(350)
No. 282	0.37	0.37	....	....	....	....	....
No. 296 Cr	Made from unfab. wire 245.						
No. 298	2.72	2.78	2.71	2.74	2.71	2.67	(397)
No. 299	3.00	3.00	....	....	....	....	....

MAKEUP OF FENCES
(See Par. B and reference characters above)

Int. Lot (Fence)	Type (d)	T-B Lot	Int. Lot (Fence)	Type (d)	T-B Lot
ZINC-COATED FENCES					
No. 510	4	No. 316	No. 410	3	....
No. 511	4	No. 317	No. 411	3	....
No. 512	4	No. 315	No. 412	3	....
No. 520	4	No. 325	No. 421	3	....
No. 521	4	No. 329	No. 422	3	....
No. 522	4	No. 327	No. 423	3	....
No. 530	4	No. 338	No. 430	3	....
No. 531	4	No. 335	No. 431	3	....
No. 532	4	No. 337	No. 432	3	....
No 540	4	No. 348	No. 433	3	....
No. 541	4	No. 345	No. 440	3	....
No. 542	4	No. 346	No. 441	3	....
No. 543	4	No. 347	No. 442	3	....
			No. 443	3	....
No. 315	2	No. 215			
No. 316	2	No. 216	No. 215	1	No. 215
No. 317	2	No. 217	No. 216	1	No. 216
No. 325	2	No. 228	No. 225	1	No. 225
No. 326	2	No. 226	No. 227	1	No. 227
No. 327	2	No. 225	No. 228	1	No. 228
No. 335	2	No. 237	No. 235	1	No. 235
No. 336	2	No. 238	No. 236	1	No. 236
No. 337	2	No. 235	No. 237	1	No. 237
No. 338	2	No. 236	No. 238	1	No. 238
No. 345	2	No. 247	No. 245	1	No. 245
No. 346	2	No. 245	No. 246	1	No. 246
No. 347	2	No. 246	No. 247	1	No. 247
No. 348	2	No. 248	No. 248	1	No. 248
No. 350	2	No. 252	No. 252	1	No. 252
No. 352	2	No. 251	No. 298	1	No. 298
No. 397	2	No. 298			
BARE-STEEL FENCES					
No. 505	4	No. 306	No. 305	2	No. 206
No. 506	4	No. 305	No. 306	2	No. 207
No. 405	3	....	No. 205	1	No. 205
No. 406	3	....	No. 206	1	No. 206

	Int. Lot	Type	T-B Lot
Coppered-covered steel fence No. 020 }	Int. No. 021	2	No. 022
Lead-coated steel fence No. 040 }	Int. No. 041	1	No. 041

UNCOATED CORROSION-RESISTANT STEEL FENCES

No. 001 (T – B Lot No. 001) Gages: Int. 12 ; T – B 12
No. 002 (T – B Lot No. 002) Gages: Int. 12.5; T – B 12.5
No. 003 (T – B Lot No. —) Gages: Int. 15 ; T – B 13

TABLE VII.—LOSS OF WEIGHTS OF ZINC-COATED WIRES.

Tests on unfabricated wires weighed and measured before exposure.
Losses of Weight in ounces per square foot

Gage of Wire	Group A[a]			Group B[a]			Group C[a]			Averages of Like Gages
	Wire Lot	Rack Exposure		Wire Lot	Rack Exposure		Wire Lot	Rack Exposure		
		West Side	East Side		West Side	East Side		West Side	East Side	
STATE COLLEGE, PA. EXPOSURE 8.29 YR.										
6........	No. 148	0.490 0.512	0.510 0.522							0.508
9........				No. 247	0.538 0.507	0.530 0.522	No. 248	0.417 0.410	0.436 0.415	0.472
11........	No. 346	0.542	0.522 0.516	No. 345	0.568 0.542	0.538 0.548	No. 348	0.440 0.440	0.436 0.421	0.501
14½.....	No. 542	0.533 0.562	0.568 0.558	No. 541	0.574 0.550	0.530 0.539	No. 540	0.440 0.409	0.399 0.393	0.505
	Average..	0.528	0.539	Average..	0.556	0.541	Average .	0.439	0.413	

[a] By reference to the published referee tests results, the wires listed under group A, (or B or C) will be found to be closely alike as regards base-metal composition and coating characteristics.

REPORT OF COMMITTEE A-6*

ON

MAGNETIC PROPERTIES

Committee A-6 on Magnetic Properties held one meeting during the past year on October 23, 1946, at Schenectady, N. Y. During the year five new members were added to the committee and there was one resignation. Since certain of the tests included in Methods A-34 involve properties other than magnetic, the scope of Committee A-6 has been revised to read, as follows:

> *Scope.*—The promotion of knowledge of the magnetic and associated properties of engineering materials and the formulation of standards relating thereto.

With reference to the Tentative Methods of Core Loss Test for Frequencies up to 2000 Cycles and Ductility Tests of Magnetic Materials (A 34 - 46 T) it was previously proposed that an investigation be made to determine how the 2000-cycle test method could be modified to make possible an increase in frequency to 10,000 cycles. It is the present consensus that if a few core loss values are determined at various frequencies from 2000 cycles down to 200 cycles, a fairly accurate extrapolation of losses up to 10,000 cycles can be made, since the relation between losses and frequency gives practically a straight line between 200 cycles and 10,000 cycles, when plotted on log-log paper.

As a result of the recent development of high-temperature insulation, particularly of the silicone type, there is a tendency to increase the normal operating temperature of the cores of electrical apparatus. At present, normal aging tests on electrical sheet are made at 100 C. The question was raised whether, in view of the above mentioned tendency, it might not be advisable to investigate aging at higher temperatures. A general discussion brought out the following observations, some of which may be based on somewhat meager data: Aging at 250 C, is no greater than at 100 C.; aging effect occurring at 100 C. may be nullified at higher temperatures; the maximum rate of aging occurs at about 130 C.; 600 hr. is too short a time to obtain the total effect; and aging is generally less at 15 kilogausses than at 10 kilogausses. On the bases of these observations, it was decided to defer the matter of a higher temperature aging test.

In the commercial testing of electrical sheet, discrepancies sometimes appear in the magnetic results between various laboratories, particularly if different types of permeameters or other equipment are used. These differences are not infrequently large enough to cause some concern since the price of electrical sheet depends on its magnetic quality. In order to assist in reducing these variations, a new Subcommittee V on Coordinating Magnetic Testing Equipment and Methods has been appointed to obtain standard samples and make them available for comparison checks at various laboratories engaged in commercial testing.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

I. NEW TENTATIVES

Proposed Tentative Method of Test for Permeability of Paramagnetic Materials.[1]—The committee recommends this method for publication as tentative. This method was originally prepared by Subcommittee VIII on Metallic Materials for Radio Tubes and Incandescent Lamps of Committee B-4, which felt the need of a simple inexpensive test to determine whether certain materials were sufficiently non-magnetic for use in some type of electron tubes. After considering a number of known methods, Howard J. Evans of the Radio Corporation of America developed a modification of the Gouy method, which seems to meet the requirements of accuracy and simplicity. It consists in weighing the sample under test in a non-uniform magnetic field. After calibration a simple calculation makes possible the determination of the permeability which may range from 1.05 to 1.0005.

Proposed Tentative Specifications for Flat-Rolled Electrical Steel.[1]—At the present time there are available no national standard procurement specifications for electrical sheet, although many companies write such specifications. The form and substance vary widely among different users. Committee A-6 has undertaken, therefore, to formulate a standard general specification for electrical sheet, which will serve as a guide for those who need to write such documents. To this there is appended an example of an individual specification which covers a specific type of sheet. The use of the general specification makes possible a very short single page individual specification and eliminates much duplication. Copies of both documents are required by the supplier. No specific values are given in the individual specification, since this is a matter for negotiation between the manufacturer and the purchaser. For special applications there may also be requirements beyond those indicated in the proposed specifications, such as special surface treatments, or closer than normal dimensional tolerances. The committee, therefore, recommends these specifications for publication as tentative.

II. ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Methods of Core Loss Test for Frequencies up to 2000 Cycles and Ductility Tests of Magnetic Materials (A 34 – 46 T)[2] be approved for reference to letter ballot of the Society for adoption as standard since no adverse comments have been received with reference to these test methods.

III. EDITORIAL CHANGES IN STANDARDS

The committee recommends as an editorial change that footnote 4 in Section 12 (*c*) of the Standard Methods of Testing Magnetic Materials (A 34 – 46) be deleted.

The committee also recommends that a symbol H_{cr} to represent relaxation coercive force be added as an editorial change to the Standard Definitions Relating to Magnetic Testing (A 127 – 46).

The recommendations appearing in this report have been submitted to letter ballot[3] of the committee, the results of which will be reported at the Annual Meeting.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (P. H. Dike, chairman).—No meetings of this subcommittee have been held during the year, but a number of suggestions for changes and additions to the magnetic definitions have been offered to the chairman. A new proposed list of International Electrotechnical letter symbols has been suggested by the United States National Committee of the International Electrotechnical Commission. A number of magnetic symbols are not in agreement with the symbols used in Standard Definitions A 127. The committee is protesting the adoption of the proposed international symbols.

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[1] These specifications and method were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-A.

[2] 1946 Book of A.S.T.M. Standards, Part I-A.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee III on Direct Current Test Methods (B.M. Smith, chairman).—Proposals have been received for alterations and additions to the permeability specifications dealing with permanent magnet materials and are under consideration. A new method for testing feebly magnetic material has been developed by this subcommittee and is recommended earlier in this report for publication as tentative. Further work is being done on the use of the 25-cm. Epstein specimen for d-c. permeability tests. It is espected that a standard method of test will result soon.

Subcommittee IV on Alternating Current Test Methods (J. P. Barton, chairman).—At the October meeting of Committee A-6, R. F. Franklin of the General Electric Co. described a machine for testing core plate enamel[4] which has been used for some time and has proved useful for quality control in the factory. He was of the opinion that it is an improvement over the stack method for interlamination resistance now given in Standard Method A 34. After general discussion, it was concluded that the two methods do not take into account the same factors and that the new method would not be a suitable substitute for the stack method. Subcommittee IV is consisidering the advisability of developing a standard covering this method.

Subcommittee V on Coordinating Magnetic Testing Equipment and Methods (J. A. Ashworth, chairman).—This new subcommittee has held one meeting since its organization. It has arranged to have a number of check samples prepared, which will be interchanged between various laboratories from time to time, in order to determine whether or not any laboratories are out of line with reference to the expected tolerances as covered by Standard Methods A 34. The National Bureau of Standards will cooperate in these tests.

This report has been submitted to letter ballot of the committee, which consists of 32 members; 28 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

THOMAS SPOONER,
Chairman.

R. L. SANFORD,
Secretary.

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[4] R. F. Franklin, "Measurement and Control of Intelaminar Resistance of Laminated Magnetic Cores," ASTM BULLETIN, No. 144, January, 1947, p. 57.

REPORT OF COMMITTEE A-7*
ON
MALLEABLE-IRON CASTINGS

At the February, 1947, meeting of Committee A-7 on Malleable-Iron Castings, it was voted to request the Board of Directors to increase the range of the functions of the committee by the inclusion of research activities, thereby broadening the rather restricted wording of the sub-heading "Scope" which is in use at present. Approval of this has been granted by the Board and plans have been made for some investigative work during the coming year which will be designed to clarify methods of testing as applied to the various types of malleable iron. Involved in this is the thought that special consideration will be given to the results in light of the definition of the terms "yield point" and "yield strength" as they appear in the Standard Definitions of Terms Relating to Methods of Testing (E 6-36) and Standard Methods of Tension Testing of Metallic Materials (E 8 - 46).

Committee A-7 very much appreciates the revision work done this past year by Committee E-1 on Methods of Testing which has resulted in simplification of these two standards. The changes which have been made are of a nature which will be helpful generally.

It is with extreme regret that the committee records the loss of the services of Harry A. Schwartz who has resigned due to pressure of business. Mr. Schwartz has been secretary of Committee A-7 for many years and has rendered valuable services both to the committee and to the Society through this period. We wish to register our sincere thanks for what he has done and are looking forward toward his continued helpfulness as a member of the committee. Mr. James H. Lansing, Consulting Engineer, Malleable Founders' Society, has consented to take over the work as secretary and has been duly elected to serve during the coming year.

RECOMMENDATIONS AFFECTING STANDARDS

During the current year the committee has reviewed the two standards and two tentatives for which it is responsible, and recommends to the Society the action indicated below. These recommendations have been submitted to letter ballot[1] of the committee, the results of which will be reported at the Annual Meeting.

I. ADOPTION OF TENTATIVE REVISIONS OF STANDARDS AS STANDARD

Standard Specifications for Malleable-Iron Castings (**A 47 - 33**),[2] as amended to include the enlarged end bar which was submitted as a tentative revision[3] in June, 1944, together with several new revisions in the text, are recommended for adoption as standard to cover malleable iron for use in castings of the 32510 and 35018 types. This recommendation was approved at the February meeting of the committee and has been confirmed by a subsequent letter ballot.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

[2] These revised specifications were accepted by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-A.

[3] 1946 Book of A.S.T.M. Standards, Part I-A, p. 1152.

Standard Specifications for Cupola Malleable Iron (**A 197 - 39**),[2] also amended to include the enlarged end bar which was submitted as a tentative revision[3] in June, 1944, together with a few revisions in the text, are recommended for adoption as standard to cover cupola type iron. This recommendation was approved at the February meeting of the committee, which action has been confirmed by a subsequent letter ballot.

II. REVISION OF TENTATIVE

Tentative Specifications for Pearlitic Malleable-Iron Castings (**A 220 - 44 T**) has been materially revised in order to meet in the best possible manner the ideas of those interested in pearlitic type malleable grades, particularly by those of The Society of Automotive Engineers and others who are concerned with the applications of these grades for use in connection with localized hardening. These specifications as now amended[2] are recommended by the committee for a trial period of two years as tentative.

III. TENTATIVE CONTINUED AS TENTATIVE WITHOUT REVISION

Tentative Specifications for Malleable Iron Flanges, Pipe Fittings, and Valve Parts (**A 277 - 44 T**) has been a recurring topic for discussion on the part of the committee, with the long-range opinion being quite equally divided as to its value, particularly as now worded. The producer interests are inclined to favor a change of the wording while the consumers generally consider the text of the present Tentative as being in accord with their desires. A recent poll of the membership, which through a misunderstanding was not taken in full accord with the intent of the February vote and therefore may not be a true gage of present opinion, showed 16 in favor of the present form and for its continuation as tentative for an additional two-year period, 20 indicated that they were in favor of a change in Section 3 (*b*), which is the principle point of criticism, two did not vote and one was neutral, this member being willing to accept the present or amended form. In the consumer group of members, eight voted in favor of the present text and one for the revision.

These specifications received further consideration at the June, 1947, meeting of the committee and a special committee has been appointed which will endeavor to develop a solution during the coming twelve months. In view of the foregoing, Committee A-7 recommends the withholding of any action for the present on specifications A 277.

This report has been submitted to letter ballot of the committee, which consists of 46 voting members; 38 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. A. KENNEDY,
Chairman.

J. H. LANSING,
Secretary.

REPORT OF COMMITTEE A-10*
ON
IRON-CHROMIUM, IRON-CHROMIUM-NICKEL, AND RELATED ALLOYS

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys held a meeting in Philadelphia, Pa., on February 26, 1947.

Two members of the committee resigned during the year and other changes in personnel occurred. The committee consists of 90 members, of whom 39 are classified as producers, 36 as consumers, and 15 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1946 Annual Meeting, Committee A-10 presented to the Society through the Administrative Committee on Standards recommendation that the following two new specifications be accepted for publication as tentative:

Tentative Specifications for:

Corrosion Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 - 46 T), and
Heat Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 297 - 46 T).

These recommendations were accepted by the Standards Committee on December 31, 1946, and the new tentatives appear in the 1946 Book of A.S.T.M. Standards, Part I-A.

NEW TENTATIVES

The committee recommends for publication as tentative new Specifications for Chromium-Nickel Corrosion Resisting Spring Wire,[1] and Corrosion-Resisting Billets and Bars for Reforging.[1]

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following four tentatives be adopted as standard:

Tentative Specifications for:

Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 - 44 T),
Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 44 T),
Seamless and Welded Austenitic Stainless Steel Tubing for the Dairy and Food Industry (A 270 - 44 T), as revised, and
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 46 T).

These specifications were originally prepared in cooperation with Committee A-1 on Steel and are under the joint jurisdiction of the two committees. Both committees are recommending the adoption as standard of the above four specifications with revisions only in A 270, the detailed changes being given in the report of Committee A-1.[2]

WITHDRAWAL OF STANDARDS

The committee recommends that the following four standards be withdrawn:

Standard Specifications for:

20 per cent Chromium, 9 per cent Nickel, Alloy-Steel Castings (A 198 - 39),
Chromium Alloy-Steel Castings (A 221 - 39),

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] These specifications were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-A.

[2] See p. 93.

Chromium-Nickel Alloy-Steel Castings (A 222 - 39), and
Nickel-Chromium Alloy-Steel Castings (A 223 - 39).

These have now been replaced by Specifications A 296 - 46 T and A 297 - 46 T referred to earlier in this report.

The recommendations appearing in this report were submitted to letter ballot[3] of the committee, the results of which will be reported at the Annual Meeting.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Classification of Data (F. P. Peters, chairman, R. Franks, secretary) has a special group consisting of F. L. LaQue and R. Franks, working in conjunction with the Alloy Castings Institute through its technical secretary, E. A. Schoefer, which has compiled a set of tables covering the properties of cast heat- and corrosion-resisting alloys. These tables have been referred to members of Committee A-10 and approved by letter ballot of the committee. Certain changes, suggested by members of the committee are to be made prior to publication. Publication of this information by the Society is now recommended as companion tables to those formerly prepared by Committee A-10 covering "Tables of Data on Chemical Compositions, Physical and Mechanical Properties of Wrought Corrosion-Resisting and Heat-Resisting Chromium and Chromium-Nickel Steels."

Subcommittee IV on Methods of Corrosion Testing (F. L. LaQue, chairman) is working out some details and modification of the program for the atmospheric exposure of corrosion-resisting steels as suggested by members of the whole committee to whom the program was sent for review and suggestions.

Subcommittee VI on Metallography (R. Franks, chairman) has been working on a program leading to the development of metallographic methods for identification of the sigma phase in iron-chromium and iron-chromium-nickel corrosion-resisting steels including both wrought and cast materials. Samples are available for distribution to members of the subcommittee and it is expected the work will be delegated during the June meeting.

Subcommittee VIII on Specifications for Bars, Forgings, and Wire (J. K. Findley, chairman) is considering some revisions in the Tentative Specifications for Hot-Rolled and Cold-Finished Corrosion-Resisting Steel Bars (A 276 - 44 T) principally concerned with minor changes in chemical composition so that the composition limits will conform to those in the AISI Classification. The subcommittee prepared and recommended acceptance by the committee of the two new tentative specifications for spring wire and bars for reforging which are referred to earlier in this report.

Subcommittee IX on Specifications for Flat Products (H. A. Grove, chairman) is studying suggestions received that the corrosion-resisting steels AISI Types 316-Cb, 317, and 405 be included in Specifications A 240 for flat products. It is also considering the substitution of a flat 8-in. specimen of full section of plate over $\frac{1}{2}$ in. in thickness as an alternate for the 2-in. gage length round test specimen presently required by Specifications A 167, A 176, and A 240.

Subcommittee X on Specifications for Castings (J. J. Kanter, chairman) prepared the two general specifications for heat- and corrosion-resisting castings mentioned earlier in this report. It has considered in connection therewith certain comments which were received and resolved at a special subcommittee meeting held during November, 1946. As indicated, this subcommittee recomments withdrawal from the Society's standards of the earlier castings specifications prepared by the subcommittee.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee XI on Specifications for Tubular Products (J. J. B. Rutherford, chairman), working jointly with Committee A-1, recommends adoption as standard of Specifications A 268, A 269, A 270, and A 271. Some modifications have been made in Specifications A 270 relating to change in title and removal of nonpolished tubing on the basis that it did not constitute sanitary tubing.

Specifications for stainless pipe are being prepared incorporating the various austenitic stainless alloys so as to permit their removal from present A.S.T.M. Specifications A 158 to a separate specification covering stainless grades of material only. This specification is being developed in cooperation with Committee A-1's Subcommittee IX, Section on Tubing, and Subcommittee XXII, Section on Piping for High-Temperature Service. Draft copies of the specification have been forwarded to interested parties for comment prior to vote in the committee.

This report has been submitted to letter ballot of the committee, which consists of 90 members; 78 members returned their ballots, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

H. D. NEWELL,
Secretary.

REPORT OF COMMITTEE B-1*
ON
WIRES FOR ELECTRICAL CONDUCTORS

Committee B-1 on wires for Electrical Conductors held three meetings during the year; two in Buffalo, N. Y., on June 25 and 26, 1946, and one in New York, N. Y. on January 27, 1947.

The committee now consists of 64 members, of whom 25 are classified as consumers, 30 as producers and 9 as general interest members.

During the year this committee has been organized into permanent standing subcommittees and the membership has been augmented to handle the new scope of activities authorized by the Society during the past year.

These new subcommittees, together with their chairmen, are as follows:

ADVISORY COMMITTEE

J. H. Foote, chairman
W. R. Hibbard
E. H. Kendall
C. S. Gordon
Alfred Bellis
H. H. Stout, Jr.
L. H. Winkler
C. E. Ambelang
P. V. Faragher
J. R. Becker
G. H. Harnden
W. F. Markley
R. S. Pratt
B. J. Sirois

SUBCOMMITTEES

Subcommittee	Chairman
I. Editorial and Records	E. H. Kendall
II. Methods of Test and Sampling Procedure	C. S. Gordon
III. Rods for Subsequent Processing	Alfred Bellis
IV. Conductors of Copper and Copper Alloys	H. H. Stout, Jr.
V. Conductors of Ferrous Metals	L. H. Winkler
VI. Composite Conductors of Copper and Steel	C. E. Ambelang
VII. Conductors of Light Metals	P. V. Faragher

This reorganization contemplates the preparation of new wire conductor specifications for materials other than copper, including aluminum, copper, steel, and composite conductors of these materials; new specifications for copper annular conductors and for rods of aluminum, steel, and copper-covered steel intended to be processed into wire for electrical conductors.

I. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions in three standards and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

These revisions largely concern the methods of gaging diameters to make more practical the requirements. These changes recognize that present day wire-drawing practices and equipment reduce the importance of diameter gaging and also render portions of spooled wire somewhat inaccessible.

Standard Specifications for Hard-Drawn Copper Wire (**B 1 – 40**),[1] and *for Medium-Hard-Drawn Copper Wire* (**B 2 – 40**),[1]

Section 3 (*b*).—Change to read as follows: "(*b*) Tests on a specimen containing a joint shall show at least 95 per cent of the minimum tensile strength given in Table I. Elongation tests shall not be made on a specimen containing a joint."

Section 5 (*b*).—Change to read as follows by the addition of the italicized words and figures:

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] 1946 Book of A.S.T.M. Standards, Part I-B.

(*b*) Within the range of diameters included in Table I the wire shall not vary from the specified diameter by more than plus or minus 1 per cent. *However, a coil or spool shall be rejected if, the measured diameter at two points being within the prescribed limits, the measured diameter at the third point (Paragraph (c)) departs from the nominal diameter by more than 2 per cent in the case of wire 0.064 in. or larger in nominal diameter, or more than 3 per cent in the case of wire under 0.064 in. in nominal diameter.*

Section 5 (c).—Change to read as follows: "(*c*) Ten per cent, but not less than five coils or spools (or all if the lot is less than five) from any lot of wire shall be gaged at three places. If accessible, one gaging shall be taken near each end and one near the middle. If any of the selected coils or spools fail to conform to the requirements prescribed in Paragraph (*b*), all coils or spools shall be gaged in the manner specified.

Section 7.—Change to read as follows: "7. No joints shall be made in the completed wire (Note). Joints in wire and rods, prior to final drawing, shall be made in accordance with the best commercial practice and shall conform to the requirements prescribed in Section 3 (*b*)."

Standard Specifications for Hard-Drawn Copper Alloy Wires (**B 105 - 39**):[1]

Section 5 (b).—Make the same change in this section as recommended above in Section 3 (*b*) of Specifications B 1.

Section 7 (b).—Add the following to this section: "However, a coil or spool shall be rejected if, the measured diameter at two points being within the prescribed limits, the measured diameter at the third point departs from the nominal diameter by more than 4 per cent over or 2 per cent under in the case of wire 0.0571 in. and larger in diameter, or by more than plus or minus 0.0015 in. (plus or minus 1.5 mils) in the case of wire having smaller nominal diameters."

Section 7 (c).—Revise this section to read as recommended above in Section 5 (*c*) of Specifications B 1.

Section 9.—Change to read as follows: "9. No joints shall be made in the completed wire (Note 7). Joints in wire and rods, prior to final drawing, shall be made in accordance with the best commercial practice and shall conform to the requirements prescribed in Section 5 (*b*).

Explanatory Notes.—Add a new Note 7 to read as follows: "NOTE 7.—Mechanical joints made during inspection at the request of the purchaser are permissible if agreed upon at the time of placing the order."

II. REVISION OF TENTATIVE

The committee recommends a revision in one tentative as follows:

Tentative Specifications for Rope-Lay-Stranded Copper Conductors Having Concentric-Stranded Members, for Electrical Conductors (**B 173 - 45 T**):[1]

Table II.—For No. 9, A.w.g. size, class H conductors, add "133" for the number of wires, "0.149 in." for outside diameter, and "41" for the weight lb. per 1000 ft.

III. CONTINUATION OF TENTATIVES WITHOUT REVISION

The committee recommends that the following tentatives be continued as tentative without revision:

Tentative Specifications for:

Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members, for Electrical Conductors (B 172 - 45 T),

Bunch-Stranded Copper Conductors for Electrical Conductors (B 174 - 45 T),

Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (B 189 - 45 T), and

Tentative Method of:

Test for Resistivity of Copper and Copper-Alloy Electrical Conductors (B 193 - 45 T).

The recommendations in this report have been submitted to letter ballot[2] of the committee, the results of which will be reported at the Annual Meeting.

This report has been submitted to letter ballot of the committee, which consists of 64 members; 51 members returned their ballots of whom 49 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. H. FOOTE,
Chairman.

E. H. KENDALL,
Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

REPORT OF COMMITTEE B-2*
ON
NON-FERROUS METALS AND ALLOYS

Committee B-2 on Non-Ferrous Metals and Alloys held one meeting on June 27, 1946, in Buffalo, N. Y., during the Annual Meeting of the Society. Since the June meeting, the work of Committee B-2 has been handled by correspondence.

The subcommittees having work for consideration held meetings at the time of the Buffalo meeting and on later dates. Business of the subcommittees has been handled by task groups and by correspondence.

At the June meeting, William E. Milligan was elected chairman of Subcommittee I, Sidney Rolle, of Subcommittee II, and O. B. J. Fraser, of Subcommittee VII. Membership changes were approved and reports of subcommittees accepted.

At Buffalo, following the meeting of Committee B-2, work on the preparation of a classification of coppers was transferred from Subcommittee I to a joint committee to which members were appointed from Committee B-2, Committee B-1 on Wires for Electrical Conductors, and Committee B-5 on Copper and Copper Alloys, Cast and Wrought. This Joint Committee on Classification of Copper met in New York, N. Y., on August 22, 1946, elected Sidney Rolle chairman, and, after review of the problem, designated a task group to prepare the classification. The task group held several meetings and has prepared a classification which will be presented to the joint committee at the June, 1947, meeting.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1946 Annual Meeting, Committee B-2 presented the following three new tentatives to the Society through the Administrative Committee on Standards, which were accepted on November 25, 1946:

Tentative Specifications for:
Fire-Refined Copper for Wrought Alloys (B 216 – 46 T),
White Metal Bearing Alloys (B 23 – 46 T), and
Soft Solder Metal (B 32 – 46 T).

The first of these specifications replaced the former Emergency Specifications for Fire-Refined Copper for Wrought Products and Alloys (ES – 7) which was withdrawn at the same time. This was recommended by Subcommittee I, which also had recommended the cancellation of Standard Specifications for Fire-Refined Copper Other Than Lake (B 72 – 33), but on letter ballot vote in the committee it was decided to retain Standard B 72. The Solder and Babbitt Specifications (B 32 and B 23) were revisions of existing specifications prepared by Subcommittee III. All three tentatives appear in the 1946 Book of A.S.T.M. Standards, Part I-B.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Specifications for Oxygen-Free Electrolytic Copper Wire Bars, Billets, and Cakes (B 170 – 44 T) be approved for reference to letter ballot of the Society for adoption as standard, without revision.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

This recommendation has been submitted to letter ballot of the committee.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Refined Copper (William E. Milligan, chairman) recommended that Tentative Specifications B 170, previously mentioned, be adopted as standard.

Work on revision of Tentative Specifications B 216 and Standard Specifications B 72 and correlation of these specifications with commercial practice and with certain specifications for fabricated copper have been completed by a task group. The revised specifications are now out to letter ballot of the subcommittee.

Subcommittee II on Refined Lead, Tin, Antimony, and Bismuth (Sidney Rolle, chairman) appointed a task group to revise the provisions for sampling pig lead in the Standard Specifications for Pig Lead (B 29 – 43). This task group expects to present its recommendations at the June, 1947, meeting.

In Buffalo reports of the survey as to the necessity for specifications for tin and specifications for antimony were considered. It was decided to defer action on any specifications for tin until the supply is more nearly normal. A recommendation for a further survey of the tin situation is to be acted upon at the next annual meeting. The survey on antimony established the need for specifications and a task group which is preparing a tentative, made a progress report at the February meeting. The group expects to make further recommendations as to these specifications at the next meeting of the subcommittee.

Subcommittee III on White Metals and Alloys (G. H. Clamer, chairman), completed during the year the revisions in the Solder and Babbit Specifications (B 32 and B 23), previously mentioned, and will meet in June to canvass the need, if any, of any further revisions therein.

Subcommittee IV on Refined Zinc and Wrought Zinc (E. H. Bunce, chairman), recommended in June, 1946, after a survey of both producers and consumers, that no need exists for specifications for mechanically grained lithographic plate.

The subcommittee has under consideration by letter ballot certain recommendations for the revision of the sampling procedure and methods of analysis in the Standard Specifications for Slab Zinc (B 6 – 46). These recommendations are in line with current A.S.T.M. practices.

Subcommittee V on Precious Metals and Alloys (R. H. Leach, chairman) met in Philadelphia, Pa., on February 28, 1947, to discuss revision of the Standard Specifications for Silver Solders (B 73 – 29).

The subcommittee is considering what action may be taken as to specifications for clad metals, such as gold-filled or rolled-gold plate.

The work of the Joint Committee on Filler Metal and the work of the American Welding Society may be of interest to the committee in the possible requirements for specifications for silver brazing alloys.

Subcommittee VI on Coated Metals (Griffith Williams, Jr., chairman) was reactivated in June and met in Atlantic City, N. J., on November 20, 1946, electing Mr. Williams chairman. At this meeting revision of the Standard Specifications for Lead-Coated Copper Sheets (B 101 – 41) was discussed and a task group appointed to prepare recommendations for chemical requirements for coatings, any changes in classification under weight of coating, and a test for continuity of coating. The work of the task group will be reviewed at the next meeting.

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[1] The letter ballot on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee VII on Refined-Nickel and High-Nickel Alloys, Cast and Wrought (O. B. J. Fraser, chairman) held a meeting in Philadelphia, Pa., on February 28, 1947, to consider proposed revision of the specifications under its jurisdiction. The meeting dealt with the Tentative Specifications for Nickel-Copper Alloy Plate, Sheet and Strip (B 127 – 41 T) and the proposed revised specifications are now being considered by letter ballot in the subcommittee. Possible revisions of other of its specifications are scheduled for discussion in June.

Subcommittee VIII On Miscellaneous Metals and Alloys (E. E. Schumacher, chairman) was requested in June, 1946, to determine the need for specifications for cadmium. The subcommittee made letter inquiry of industry, producers, and consumers. As a result of their inquiry a meeting was held in Philadelphia, Pa., on February 27, 1947, which was attended by representatives of producers and representatives of the several classes of consumers, alloy makers, bearing makers, and platers. After a short discussion in which no real desire for specifications was expressed by the consumers, it was voted that no need for specifications for cadmium exists.

This report has been submitted to letter ballot of the committee which consists of 125 members; 93 members returned their ballots, of whom 90 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. E. THUM,
Chairman.

G. H. LEFEVRE,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-2 presented to the Society through the Administrative Committee on Standards a revision of the Tentative Specifications for Fire-Refined Copper for Wrought Alloys (B 216 – 46 T) which included a change in title to read, "Tentative Specifications for Fire-Refined Copper for Wrought Products and Alloys." The Committee also submitted a recommendation for revision and reversion to tentative of the Standard Specifications for Fire-Refined Copper Other than Lake (B 72 – 33) in the form of Tentative Specifications for Fire-Refined Casting Copper (B 72 – 47 T).

These recommendations were accepted by the Standards Committee on September 4, 1947, and the new and revised tentatives appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

REPORT OF COMMITTEE B-3*

ON

CORROSION OF NON-FERROUS METALS AND ALLOYS

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held meetings in Buffalo, N. Y., on June 26, 1946, and in Philadelphia, Pa., on February 26, 1947.

The committee consists of 90 members, of whom 77 are voting members; 36 are classified as producers, 28 as consumers, and 13 as general interest members.

EDITORIAL CHANGE IN TENTATIVE METHOD

The committee voted to continue as tentative the Method of Alternate Immersion Corrosion Testing of Non-Ferrous Metals (B 192 - 44 T), with an editorial change in Section 9 (*a*). It is proposed to add the following sentence at the end: "Loss in weight measurements will not show the true corrosion rate if metals are severely pitted or selectively corroded."

CONTINUATION OF TENTATIVES WITHOUT REVISION

The committee also voted to continue as tentative without revision the Method of Total Immersion Corrosion Test of Non-Ferrous Metals (B 185 - 43 T). and the Method of Salt Spray (Fog) Testing (B 117 - 44 T).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Total Immersion Test (A. W. Tracy, chairman) has formed a subgroup to carry out cooperative tests to determine whether Tentative Method B 185 will give consistent results.

Subcommittee II on Alternate Immersion Test (H. L. Burghoff, chairman) has postponed its cooperative test program until the proposed program of Subcommittee I has been completed.

Subcommittee IV on Humidity Test (L. J. Waldron, chairman) held its organization meeting in February, 1947, when reports on the present status of humidity testing were presented by several members. These reports will be circulated among the subcommittee members for study prior to formulating any plan of work.

Subcommittee VII on Weather (F. L. LaQue, chairman) has been active through a subgroup on instrumentation headed by O. B. Ellis. This subgroup held a meeting on February 26, 1947, and discussed weather data, such as are available from weather bureau stations, and the type of instruments which may be available for measuring weather variables that might be expected to have some bearing on corrosion rates. It was the consensus of the committee that weather bureau data would not be useful for work of this type. The weather bureau observations are made at points remote from the corrosion testing stations and do not afford the specific information required.

Recording instruments for the measurement of temperature, rainfall, wind direction, wind velocity, and total solar radiation are readily available. There is some doubt as to the accuracy of any of the recording instruments for atmospheric humidity. There is some question as to the availability of any instrument for the accurate measurement and recording of atmospheric pollution.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

Members of the committee are continuing observations on the functioning of these instruments in the hope of formulating a program for the correlation of weather observations and corrosion rates of certain key materials.

Subcommittee VIII on Galvanic and Electrolytic Corrosion (L. J. Gorman, chairman).—A set of stainless steel couples, which has been on exposure five years, was taken in during the summer of 1946, tested for contact resistance, disassembled, and visually inspected. The data will be reported when weight changes have been determined.

Galvanic corrosion tests on magnesium coupled with various other metals and alloys are being assembled and it is expected the couples will be exposed during the summer.

This report has been submitted to letter ballot of the committee, which consists of 77 voting members; 62 members returned their ballots, of whom 59 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. S. RAWDON,
Chairman.

A. W. TRACY,
Secretary.

REPORT OF COMMITTEE B-4[*]

ON

ELECTRICAL HEATING, RESISTANCE, AND RELATED ALLOYS

Committee B-4 on Electrical Heating, Resistance, and Related Alloys held three meetings since its last report: in Atlantic City, N. J., on May 15 to 17 and October 30 to November 1, 1946, inclusive, and on February 26 to 28, 1947, in Philadelphia, Pa., in conjunction with the Spring Group Meetings of A.S.T.M. Committees.

The committee consists of 69 members of whom 28 are classified as producers, 35 as consumers, and 6 as general interest members.

By action of the Advisory Committee confirmed by the Board of Directors, the title of Committee B-4 was changed from "Electrical-Heating, Electrical-Resistance, and Electric-Furnace Alloys" to the simplified one given in the above heading.

I. New Tentatives

The committee recommends that the two new proposed methods: Measuring Residual Stress in Cylindrical Metal-to-Glass Seals[1] and Testing Fine Round and Flat Wire for Electronic Devices,[1] the latter being a consolidation and revision of Tentative Methods B 118 and B 156, be accepted for publication as tentative.

II. Withdrawal of Tentatives

The committee recommends that the Tentative Methods of Testing Nickel and Nickel-Alloy Wire and Ribbon for Electronic Tube Filaments (B 118 - 42 T)[2] and Testing Lateral Wire for Grids of Electronic Devices (B 156 - 42 T)[2] be withdrawn.

The recommendations appearing in this report have been submitted to letter ballot[3] of the committee, the results of which will be reported at the Annual Meeting.

Activities of Subcommittees

Subcommittee II on Electrical Tests (P. H. Dike, chairman) is planning action upon the Standard Method of Test for Resistivity of Metallic Materials (B 63 - 36) after a special task group of Committee B-1 on Wires for Electrical Conductors has made recommendations regarding a proposed revision of the Tentative Method of Test for Resistivity of Copper and Copper-Alloy Electrical conductors (B 193 - 45 T) which, it is expected, will coordinate the two methods.

Subcommittee V on Wrought and Cast Alloys for High-Temperature Use (J. W. Harsch, chairman) is revising the Tentative Specifications for Nickel-Chromium-Iron Alloy Castings (35-15 Class) for High-Temperature Service (B 207 - 46 T) in order to clarify the specifications and also to include the "38-18 class." This revision will be submitted for letter ballot at a later date for action by the Administrative Committee on Standards.

Subcommittee VII on Thermostat Metals (P. H. Brace, chairman) is still investigating test methods for determining the comparative performance of mounted thermostat elements. Microhardness test methods are also being pursued and a check is being made on lack of agreement under very light loads.

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] These methods were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-A.

[2] 1946 Book of A.S.T.M. Standards, Part I-B.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee VIII on Metallic Materials for Radio Tubes and Incandescent Lamps (S. A. Standing, chairman) has been very active throughout the year. The various subsections of Section A on Cathode Materials have been working on the following projects: Development of a figure of merit for cathode melts and establishment of an improved form of reporting; check analyses of a number of heats of cathode nickel and preparation of report sheets to be used by laboratories making analyses; investigation of the identification of nonmetallics and oxides present in cathode material, which, combined with electrical resistivity determinations, may be helpful in estimating the emission activity of cathode materials; and consideration of revisions in the Standard Methods of Testing Sleeves and Tubing for Radio Tube Cathodes (B 128 – 42).

Section B is working with Committee A-6 on Magnetic Properties on the development of a method for evaluation of magnetic permeability of slightly magnetic materials.

Section D is continuing the study of particle size and density and is investigating a statistical approach. A joint meeting with a representative of Committee E-11 on Quality Control of Materials is being planned. A statistical method should greatly simplify the tests and shorten the time consumed therefor.

Section E prepared the two new methods of test referred to at the beginning of this report.

Subcommittee IX on Methods of Test for Alloys in Controlled Atmospheres (P. H. Brace, chairman) is working on an investigation of the causes and prevention of "green rot" in electric furnaces using controlled atmospheres.

Subcommittee X on Contact Materials (F. E. Carter, chairman) has also been very active during the year. Section A on Life Tests is continuing its investigation of electrical characteristics of contacts. While present studies have been based upon operations at relatively low currents, it is probable that the committee may be asked to extend the tests to much higher currents. Section B on Physical Properties is studying methods of measuring electrical conductivity, temperature coefficient of electrical conductivity, and density of metallic contacts. It has been agreed for the present to standardize on a smooth machine finish on test contacts.

Section E on Bibliography prepared the 1946 Supplement to the Bibliography and Abstracts on Electrical Contacts which is now available as a separate publication.

This report has been submitted to letter ballot of the committee, which consists of 69 members; 39 members returned their ballots, of whom 36 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

JOHN W. HARSCH,
Chairman.

F. E. BASH,
Secretary.

REPORT OF COMMITTEE B-5*
ON
COPPER AND COPPER ALLOYS, CAST AND WROUGHT

Committee B-5 on Copper and Copper Alloys, Cast and Wrought, held two meetings during the year at Washington, D. C., on September 19, 1946, and at Philadelphia, Pa., on February 28, 1947. At these sessions the Advisory Committee and Subcommittees A-1, A-2, A-3, A-4, C-1 and C-3 also met.

During the year 7 members were added to the committee and there were 11 removals. At the present time the committee consists of 132 members, of whom 112 are voting members; 49 are classified as producers, 46 as consumers, and 17 as general interest members.

Recommendations Affecting Standards

The committee is submitting one new tentative method, revisions in 25 tentative specifications, and revisions in 20 standards for immediate adoption. The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.

The recommendations in this report have been submitted to letter ballot[1] of the committee, the results of which will be reported at the Annual Meeting.

Activities of Subcommittees

The following is the report of the several subcommittees during the year. The actions are given in detail in the Appendix.

Subcommittee A-1 on Plate, Sheet, and Strip (R. A. Frye, chairman, S. A. Rosecrans, secretary) recommended that revisions be incorporated in eleven tentative specifications: B 19, B 36, B 121, B 122, B 129, B 130, B 131, B 152, B 169, B 194, and B 195. Revisions for immediate adoption were recommended in six standard specifications: B 11, B 96, B 97, B 100, B 103, and B 171.

A task group (G. R. Gohn, chairman) prepared the revisions in chemical limits for Specifications B 194 and B 195, and also prepared similar limits for Specifications B 196 (Rod) and B 197 (Wire). The transfer of all but annealed material from Specifications B 96 to B 97 was prepared by a task group with J. E. McGraw as chairman. A task group (V. P. Weaver, chairman) prepared the revisions for annealed tempers in Specifications B 36. Another task group (N. H. Murdza, chairman) prepared the revisions for Specifications B 129 and B 131. A task group (J. D. MacQueen, chairman) prepared the revisions in manufacturing requirements in plate and strip specifications. A task group (N. H. Murdza, chairman) prepared plate requirements to be added to sheet and strip specifications.

The subcommittee has under way the revision of requirements for mechanical properties, Specifications B 194 and B 195.

Subcommittee A-2 on Rods, Bars, and Shapes (H. C. Ashley, chairman) recommended that revisions be incorporated in seven tentative specifications: B 21, B 133, B 139, B 150, B 151, B 187, and B 196. Revisions for immediate adoption were recommended in six standard specifications: B 12, B 16, B 98, B 124, B 138, and B 140.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

A task group (G. Williams, Jr., chairman) prepared some of the revisions of tensile properties for Specifications B 16. A task group (H. S. Freynik, chairman) prepared the revised Specifications B 151.[2] A task group (M. J. Mianulli, chairman) prepared the proposed method for measuring straightness of rod.

Among the suggestions being studied by the subcommittee are the broadening of the scope of Specifications B 133 and B 187, the possible deletion of the bend test in the rod and bar specifications, and the application of the new proposed Tentative Methods of Tension Testing of Copper and Copper-Alloy Rods, Bars and Shapes.[3]

Subcommittee A-3 on Wire and Wire Rod (B. J. Sirois, chairman) recommended that revisions be incorporated in three tentative specifications: B 159, B 197, and B 206. Revisions for immediate adoption were recommended in Standard Specifications B 99 and B 134.

A task group (A. E. Drobish, chairman) prepared the revisions in physical properties for Specifications B 99 and B 159. A task group (G. Williams, Jr., chairman) prepared the revisions in physical properties for Specifications B 134. A task group (H. S. Freynik, chairman) prepared the revisions in physical properties for Specifications B 197. A task group (H. S. Freynik, chairman) prepared the revisions in physical properties for Specifications B 206.

The subcommittee is preparing further revisions in physical properties for Specifications B 197.

Subcommittee A-4 on Pipe and Tube (Alan Morris, chairman, R. S. Pratt, secretary) recommended that revisions be incorporated in three tentative specifications: B 75, B 135, and B 188. Revisions for immediate adoption were recommended in five standard specifications: B 42, B 43, B 68, B 88, and B 111.

A task group (H. C. Ashley, chairman) prepared the revisions of physical properties for Specifications B 88. A task group (V. P. Weaver, chairman) prepared requirements for 90–10 copper-nickel alloy for Specifications B 111.

Among the subjects being studied by the subcommittee are the review of hydrostatic and pneumatic test requirements in pipe and tube specifications, the development of requirements for dehydrated and sealed tubes in Specifications B 68, and the incorporation of minimum values for additive elements for Admiralty metal and aluminum brass in Specifications B 111.

Subcommittee B-1 on Castings and Ingots for Remelting (G. H. Clamer, chairman, C. S. Cole, secretary) recommended that revisions be incorporated in Tentative Specifications B 22. The subcommittee has under way a revision of requirements for sampling for chemical analysis in Specifications B 30 and B 52, and is studying the subject of test bars and test bar practice.

Subcommittee C-1 on Methods of Test (G. R. Gohn, chairman, J. P. Guerard, secretary) recommended revisions for immediate adoption in Standard Method B 153 and the acceptance of the new proposed Tentative Methods of Tension Testing of Copper and Copper Alloy Rods, Bars, and Shapes[2] prepared by a task group (S. A. Rosecrans, chairman).

The subcommittee is studying the development of a bend test method for rods and bars, preparation of a standard referee clause, the effect of speed of testing on tensile properties, the relation of Rockwell hardness to thickness of specimen, and the development of methods of tension testing for thin sheet metals.

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[2] These revised specifications were accepted by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

[3] These methods were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

Subcommittee C-2 on Tolerances (J. E. McGraw, chairman), in collaboration with Subcommittees A-1, A-2, and A-3, conducted a referendum letter ballot concerning the proposed CABRA classification of flat products.

Among the subjects being studied by the subcommittee are the revision of length and straightness tolerances in Specifications B 187, the revision of diameter tolerances in Specifications B 138, and, in collaboration with CABRA, the revision of tolerances in Specifications B 111 and the development of methods for measuring the straightness of shafting and piston finish rod.

Subcommittee C-3 on Editorial and Publications (W. F. Roeser, chairman) recommended the deletion of the present note as to significance of dimensional tolerances in wrought product specifications and is studying the development of a replacing requirement. It is continuing its study of the correlation of the chemical limits and of the nomenclature and classification of wrought alloys under the jurisdiction of the committee.

This report has been submitted to letter ballot of the committee, which consists of 112 voting members; 93 members returned their ballots, of whom 81 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. H. HARNDEN,
Chairman.

V. P. WEAVER,
Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards covering copper and copper alloys which are referred to earlier in this report. The standards appear in their present form in the 1946 Book of A.S.T.M. Standards, Part I-B.

REVISIONS OF TENTATIVE SPECIFICATIONS

The committee recommends that the following 25 tentative specifications be revised as indicated below and continued as tentative.

Tentative Specifications for Cartridge Brass Sheet, Strip, Plate, Bar, and Disks (B 19 - 46 T):

Section 3.—Revise Paragraph (*a*) to read as follows: "The material shall be of such quality and purity that the finished product shall have the properties and characteristics prescribed in these specifications."

Delete Paragraph (*b*) and reletter Paragraph (*c*) as (*b*).

Section 7.—Delete references to B. & S. gage numbers and percentage reduction.

Section 13.—Revise to read as follows:

13. *Methods of Test and Chemical Analysis.*—(*a*) The properties and chemical compositions enumerated in these specifications shall, in case of disagreement, be determined in accordance with the following methods of the American Society for Testing Materials:

Test	A.S.T.M. Designation[a]
Chemical Analysis...............	E 36[b]
Tension.........................	E 8[c]
Grain Size......................	E 2, E 3
Rockwell Hardness...............	E 18

[a] These designations refer to the following methods of testing:
Standard Methods of Chemical Analysis of Brasses (A.S.T.M. Designation E 36),
Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8),
Tentative Methods of Preparation of Micrographs of Metals and Alloys (A.S.T.M. Designation: E 2),
Tentative Methods of Preparation of Metallographic Specimens (A.S.T.M. Designation: E 3), and
Standard Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (A.S.T.M. Designation: E 18).

[b] Spectrographic analysis may be permitted for determining the amount of impurities prescribed in Section 4, subject to agreement between the manufacturer and the purchaser, or subject to chemical analysis as the basis of acceptance or rejection.

[c] The tension test specimen shall conform to the dimensions shown in Fig. 2 or Fig. 3 of Standard Methods E 8.

(*b*) *Measurement of Dimensions.*—At least ten sheets, strips, plates, bars, or disks shall be selected at random so as to be representative of the lot of material. The dimensions of each sheet, strip, plate, bar, or disk so selected shall be measured. Measurements for thickness shall be made over the entire width or diameter of the piece; and the measurements shall be made in at least three different places not less than, except for disks, 1 ft. from each end, and at such other points as may be selected.

Section 14.—Delete Note 2.

Sections 14, 15, 16, and 18.—Combine in one section under the title "Dimensional Tolerances" and renumber subsequent sections accordingly.

Table V.—Revise to read the same as Table III in Specifications B 152 - 46 T.

Tentative Specifications for Naval Brass Rods, Bars, and Shapes (B 21 - 46a T):

Section 3.—Reletter the present section as Paragraph (*b*) and add the following as Paragraph (*a*): "The material shall be of such quality and purity that the finished product shall have the properties and characteristics prescribed in these specifications."

Section 11.—Revise to read as indicated above for Section 13 (*a*) in Specifications B 19, listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 36
Tension	E 8
Mercurous Nitrate	B 154

Section 12.—Delete the note.

Section 13.—Combine with Section 12 and renumber subsequent sections accordingly.

Section 14.—Add a note to read as follows: "NOTE.—To determine compliance with this section, rods and bars other than piston finish and shafting rods shall, in case of disagreement, be checked by the following method: Place the rods or bars on a level table so that the arc or departure from straightness is horizontal. Measure the maximum depth of arc to the nearest 1/32 in., using a steel scale and a straightedge."

Tentative Specifications for Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 – 46 T):

Section 2.—Revise Paragraph (*a*) to read as follows, relettering the present Paragraph (*b*) as (*c*):

> (*a*) Each of the alloys called for in these specifications shall be made from materials such that the finished product will conform to the requirements for the specified class.
>
> (*b*) The preferred method of compounding classes A and B is the use of new metal (Note).
>
> NOTE.—Materials conforming to the following specifications are satisfactory for use in the manufacture of bronze castings described in these specifications:
>
> | Material | A.S.T.M. Designation[a] |
> |---|---|
> | Copper | B 4, B 5, B 115, or B 216 |
> | Phosphor Copper | B 52 |
> | Tin | Equal to "Banca" or "Straits" |
> | Zinc | B 6 |
> | Ingot (for class C) | B 30, Alloy 3A |
> | Ingot (for class D) | B 30, Alloy 1A |
>
> Clean new scrap of known composition, produced at the foundry at which the bronze is cast, may also be used.

Tentative Specifications for Brass Sheet and Strip (B 36 – 46 T):

Title.—Revise the title to read "Tentative Specifications for Brass Plate, Sheet, and Strip" and substitute "plate, sheet, and strip" for "sheet and strip" throughout the specifications.

Section 1 (a).—After the word "commercial" insert the word "plate."

Section 3.—Revise to read as indicated above for Section 3 in Specifications B 19.

Section 4.—In the table of chemical composition, change the maximum permissible lead content for alloy No. 6 from the present "0.05" to read "0.07" per cent.

Section 6.—Delete references to B. & S. gage numbers and percentage reduction, and revise to read as follows:[1]

> 6. The tempers of rolled plate, sheet, and strip brasses shall be designated as shown in Table I in the column titled "Rolled Temper."
>
> NOTE.—Various ready-to-finish grain sizes may be designated within the limits specified for

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[a] These designations refer to the following specifications:
Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (A.S.T.M. Designation: B 4),
Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (A.S.T.M. Designation: B 5),
Standard Specifications for Electrolytic Cathode Copper (A.S.T.M. Designation: B 115),
Tentative Specifications for Fire-Refined Copper For Wrought Alloys (A.S.T.M. Designation: B 216),
Standard Specifications for Phosphor Copper (A.S.T.M. Designation: B 52)
Standard Specifications for Slab Zinc (Spelter) (A.S.T.M. Designation: B 6), and
Tentative Specifications for Copper-Base Alloys In Ingot Form for Sand Castings (A.S.T.M. Designation: B 30).

[1] This editorial revision will be carried out in all the revised sheet and strip specifications where it is applicable when the specifications are next printed.

the several annealed tempers under Section 10, if agreed upon between the manufacturer and the purchaser. This agreement should consider only those tempers up to and including the three quarter hard.

Section 7.—Revise to read as follows:[1] "7. The tempers of annealed plate, sheet, and strip brasses shall be designated as shown in Table II in the column titled "Nominal Grain Size" and for the respective alloys are available only for the nominal grain sizes shown therein."

Table I.—Add a note to read as follows: NOTE.—Plate is generally available in only the soft, quarter hard, and half hard tempers. Required properties for other tempers shall be agreed upon between the manufacturer and the purchaser at the time of placing the order."

Table II.—For alloys Nos. 6 and 8, change the maximum grain size for 0.070-mm. nominal grain from the present "0.100" to read "0.120" mm. Revise alloys Nos. 1, 2, 3, and 4 by adding another annealed temper having 0.025-mm. nominal grain size with a minimum grain size of 0.015 mm. and a maximum of 0.035 mm.

Table III.—Revise by the addition of the following values for alloys Nos. 1, 2, 3, and 4 and the revision of values for alloys Nos. 6 and 8 as shown (values for alloys Nos. 6 and 8 are identical):

Annealed Temper, Nominal Grain Size	Rockwell Hardness (Values Included as Information Only)			
	F Scale		Superficial 30-T	
	Min.	Max.	Min.	Max.
Alloy No. 1 (95 Cu, 5 Zn): 0.025 mm.	50	61	1	17
Alloy No. 2 (90 Cu, 10 Zn): 0.025 mm.	58	70	13	31
Alloy No. 3 (85 Cu, 15 Zn): 0.025 mm.	60	72	16	34
Alloy No. 4 (80 Cu, 20 Zn): 0.025 mm.	63	77	20	42
Alloy No. 6 (70 Cu, 30 Zn) and Alloy No. 8 (65 Cu, 35 Zn): 0.070 mm.	52	67	3	27

Section 13.—Revise to read as indicated above for Section 13 (*a*) in Specifications B 19, listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 36
Tension	E 8
Grain Size	E 2, E 3
Rockwell Hardness	E 18

Section 14.—Delete Note 2.

Table IV.—Revise to read the same as Table III in Specifications B 152 - 46 T.

Sections 14, 15, 17, and 18.—Combine in one section under the title "Dimensional Tolerances" and renumber subsequent sections accordingly.

Tentative Specifications for Seamless Copper Tubes (B 75 - 46a T):

Section 3.—Revise Paragraph (*a*) to read as follows: "The material shall be copper of such quality and purity that the finished product shall have the properties and characteristics prescribed in these specifications, and shall be cold drawn to size."

Renumber Paragraph (*b*) as Section 6 under the title "Temper" and renumber subsequent sections accordingly. For drawn tempers add a reference to "drawn (general purpose)."

Section 4.—For type A change the maximum phosphorus content from the present "0.010" to read "0.012."

Section 6.—Delete Section 6 and renumber subsequent sections accordingly.

Section 14.—Retitle "Methods of Test and Chemical Analysis" and revise the introductory paragraph to read as follows, lettering as Paragraph (*a*):

> 14. (*a*) The properties and chemical compositions enumerated in these specifications shall, in case of disagreement, be determined in accordance with the following methods of the American Society for Testing Materials, with the modifications given in Paragraphs (*b*), (*c*) and (*d*):

Test	A.S.T.M. Designation
Chemical Analysis	E 53, E 62
Tension	E 8
Grain Size	E 2
Expansion (Pin Test)	B 153
Rockwell Hardness	E 18
Electrical Resistivity	B 193

Reletter the present Paragraph (*a*) as (*b*), omitting the first sentence. Reletter the present Paragraph (*b*) as (*c*), omitting the fourth sentence. Delete the present Paragraphs (*c*) and (*d*). Reletter the present Paragraph (*e*) as (*d*), omitting the first sentence.

Section 16.—Delete the note.

Table II.—Rearrange Table II to start with small wall thicknesses and avoid the abbreviation "excl." by using "incl."

Sections 16 and 17.—Combine in one section under the title "Dimensional Tolerances" and renumber subsequent sections accordingly.

Tentative Specifications for Leaded Brass Sheet and Strip (B 121 – 46 T):

Title.—Revise the title to read "Tentative Specifications for Leaded Brass Plate, Sheet, and Strip" and substitute "plate, sheet, and strip" for "sheet and strip" throughout the specifications.

Section 3.—Revise to read as indicated above for Section 3 in Specifications B 19.

Section 6.—Delete the references to B. & S. gage numbers and percentage reduction.

Table I.—Add a note to Table I to read as indicated above for Table I in Specifications B 36.

Section 13.—Revise to read as indicated above for Section 13 in Specifications B 36.

Section 14.—Delete Note 2.

Table IV.—Add thickness tolerances for plate as indicated above for Table IV in Specifications B 36.

Sections 14, 15, 17, and 18.—Combine in one section under the title "Dimensional Tolerances," and renumber subsequent sections accordingly.

Tentative Specifications for Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 – 46a T):

Title.—Revise the title to read "Tentative Specifications for Copper-Nickel-Zinc and Copper-Nickel Alloy Plate, Sheet, and Strip" and substitute "plate, sheet, and strip" for "sheet and strip" throughout the specifications.

Section 3.—Revise Paragraph (*a*) to read as indicated above for Section 3 (*a*) in Specifications B 19.

Add a new Paragraph (*b*) to read as follows: "(*b*) The material shall be finished by cold rolling and annealing or by cold rolling and may be finished in either the cold rolled or annealed conditions, as required."

Table II.—Add a note to Table II to read as indicated above for Table I in Specifications B 36.

Section 13.—Revise to read as indicated above for Section 13 in Specifications B 36, omitting "E 36" in the table.

Section 14.—Delete Note 2.

Table IV.—Revise to read the same as Table III in Specifications B 103 – 46.

Sections 14, 15 and 17.—Combine in one section under the title "Dimensional Tolerances" and renumber subsequent sections accordingly.

Appendix I.—Delete Appendix I.

Tentative Specifications for Cartridge Brass Cartridge Case Cups (B 129 – 46 T):

Section 3.—Revise to read as indicated above for Section 3 (*a*) in Specifications B 19.

Table I.—In Table I for type I material, change the values for the minimum diameter of average grain size from "0.040" and "0.080" to read "0.045" and "0.065" mm., respectively. Also change the values for the maximum diameter of average grain size from "0.100" and "0.140" to read "0.120" and "0.150" mm., respectively.

Section 7.—Retitle "Methods of Test

and Chemical Analysis" and revise the introductory paragraph to read as follows, lettering as Paragraph (*a*):

> 7. (*a*) The properties and chemical compositions enumerated in these specifications shall, in case of disagreement, be determined in accordance with the following methods of the American Society for Testing Materials, with the modification given in Paragraph (*b*):

Test	A.S.T.M. Designation
Chemical Analysis	E 36
Grain Size	E 2, E 3

Reletter the present Paragraph (*a*) as (*b*), omitting the first sentence. Reletter the present Paragraph (*b*) as (*c*). Reletter the present Paragraph (*c*) as (*d*), revising the second sentence to read as follows: "Each cup shall be gaged for compliance with all the dimensions shown on the applicable drawings."

Tentative Specifications for Gilding Metal Strip (B 130 - 46 T):

Section 3.—Revise to read as indicated above for Section 3 in Specifications B 19.

Section 4.—In the table on chemical composition change the composition range for copper from the present "89.0 to 92.0" to read "89.0 to 91.0" per cent.

Section 7.—Delete the references to B. & S. gage numbers and percentage reduction.

Section 13.—Retitle and revise the introductory paragraph to read as indicated above for Section 13 (*a*) in Specifications B 19, lettering as Paragraph (*a*).

Delete the present Paragraphs (*a*), (*b*), (*c*) and (*d*). Reletter the present Paragraph (*e*) as (*b*).

Section 14.—Delete Note 2.

Sections 14, 15 and 17.—Combine in one section under the title "Dimensional Tolerances," and renumber subsequent sections accordingly.

Tentative Specifications for Gilding Metal Bullet Jacket Cups (B 131 - 46 T):

Section 3.—Revise to read as indicated above for Section 3 (*a*) in Specifications B 19.

Table I.—Revise to read as shown in the accompanying Table I.

TABLE I.—GRAIN SIZE REQUIREMENTS ON SIDEWALL[a].

Class	Diameter of Average Grain Size, mm.		
	Nominal	Min.	Max.
I (not annealed)[b]	....	....	....
II (annealed)	0.025	0.015	0.045

[a] Approximately midway of the length of the sidewall

[b] Class I may be specified upon mutual agreement between the manufacturer and the purchaser.

Section 8.—Retitle and revise the introductory paragraph to read as indicated above for Section 7 (*a*) in Specifications B 129, lettering as Paragraph (*a*).

Delete the present Paragraph (*a*). Paragraphs (*b*) and (*c*) remain unchanged except revise the second sentence of Paragraph (*c*) to read "Each cup shall be gaged for compliance with all the dimensions shown on the applicable drawing."

Tentative Specifications for Copper Rods, Bars, and Shapes (B 133 - 46a T):

Section 7.—Delete and renumber subsequent sections accordingly.

Section 16.—Retitle and revise to read as indicated above for Section 13 (*a*) in Specifications B 19, listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 53
Tension	E 8
Rockwell Hardness	E 18

Section 18.—Delete the note.

Section 19.—Combine with Section 18 and renumber subsequent sections accordingly.

Section 20.—Add a note to read as follows: "NOTE.—To determine compliance with this section, rods and bars shall in case of disagreement, be checked by the following method: Place the rod or bar on a level table so that the arc or departure from straightness is horizontal. Measure the maximum depth of arc to the nearest 1/32 in. using a steel scale and a straightedge."

Tentative Specifications for Miscellaneous Brass Tubes (B 135 – 46a T):

Section 3.—Revise Paragraph (*a*) to read as follows: "The material shall be copper-zinc or copper-zinc-lead alloy of such quality and purity that the finished product shall have the properties and characteristics prescribed in these specifications."

Delete the present Paragraph (*b*) and reletter Paragraph (*c*) as (*b*).

Section 12.—Retitle "Methods of Test and Chemical Analysis." In the second line, after the word "shall" add the words "in case of disagreement." In the list of tests, add for "Chemical Analysis," A.S.T.M. Designation "E 36" and for "Grain Size," A.S.T.M. Designation "E 3."

Section 15.—Delete the note.

Sections 15 and 16.—Combine in one section under the title "Dimensional Tolerances" and renumber subsequent sections accordingly.

Table III.—Rearrange Table III as indicated above for Table II in Specifications B 75.

Tentative Specifications for Phosphor Bronze Rods, Bars, and Shapes (B 139 – 46a T):

Section 1.—Delete the figures and words "0.250 in. and over in diameter or thickness, and."

Section 3.—Revise to read as indicated above for Section 3 in Specifications B 19.

Table I.—Change the composition limits for phosphorus and zinc for grade B 2 to read as prescribed in the following table:

	Revised Values	Present Values
Phosphorus, per cent	0.01 to 0.50	0.03 to 0.50
Zinc, per cent	1.50 to 4.50	3.00 to 4.50

Revise footnote *a* to read as follows: "In the case of grades A, C, and D rods 1.25 in. and over in diameter, a maximum manganese content and maximum iron content of 1.25 per cent may be permitted and these two elements shall be considered as specified elements."

Section 6.—Delete and renumber subsequent sections accordingly.

Section 11.—Retitle and revise to read as indicated above for Section 13 (*a*) in Specifications B 19, listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 54
Tension	E 8

Section 12.—Delete the note.

Section 13.—Combine with Section 12 and renumber subsequent sections accordingly.

Section 14.—Revise to read as indicated above for Section 14 in Specifications B 21.

Tentative Specifications for Aluminum Bronze Rods, Bars, and Shapes (B 150 – 46a T):

Section 3.—Reletter the present section as Paragraph (*b*) and add a new Paragraph (*a*) to read as indicated above for Section 3 (*a*) in Specifications B 19.

Section 11.—Retitle and revise to read as indicated above for Section 13 (*a*) in Specifications B 19, listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 54
Tension	E 8
Mercurous Nitrate	B 154

Section 12.—Delete the note.

Section 13.—Combine with Section 12 and renumber subsequent sections accordingly.

Section 14.—Revise to read as indicated above for Section 14 in Specifications B 21.

Tentative Specifications for Copper-Nickel-Zinc Alloy Rod, Bar, and Wire (B 151 - 46a T):

The proposed revision of these specifications includes the deletion of all requirements for wire, the addition of another alloy, revision in tempers and tensile requirements, deletion of reference to B. & S. gage numbers and percentage reduction, revision in requirements for manufacture, the addition of a method for checking straightness, and the deletion of the note as to significance of dimensional tolerances. The revised specifications are appended hereto.[2]

Tentative Specifications for Copper Sheet, Strip, and Plate (B 152 - 46 T):

Section 3 (a).—Revise to read as indicated above for Section 3 (*a*) in Specifications B 19.

Section 6 (a).—Delete the references to B. & S. gage numbers and percentage reduction.

Section 16.—Retitle and revise to read as indicated above for Section 13 (*a*) in Specifications B 19, listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 53, E 62
Tension	E 8
Grain Size	E 2, E 3
Rockwell Hardness	E 18
Electrical Resisitivity	B 193

Section 17.—Delete Note 2.

Sections 17, 19, and 21.—Combine in one section under the title "Dimensional Tolerances," and renumber subsequent sections accordingly.

Tentative Specifications for Phosphor Bronze Wire (B 159 - 46a T):

Section 3.—Revise to read as follows: "The material shall be phosphor-bronze alloy of such quality and purity that the finished product shall have the properties and characteristics prescribed in these specifications."

Section 5.—Revise to read as follows:

> 5. (*a*) Pieces from four individual coils shall be taken for each lot of 5000 lb. or fraction thereof.
>
> (*b*) Drillings, millings, or clippings for chemical analysis shall be taken from the representative sample selected for the purpose of tests. Equal quantities shall be taken from each sample and mixed thoroughly.
>
> (*c*) The minimum weight of the sample required for analysis is 150 g. The drillings, millings, or clippings shall be divided into three equal parts, each of which shall be placed in a sealed container, one for the manufacturer, one for the purchaser, and one for an umpire, if necessary.
>
> (*d*) In case of dissatisfaction, either party may require that the material be resampled in the presence of representatives of the manufacturer and the purchaser. The thoroughly mixed sample shall be divided into three equal parts, each of which shall be placed in a sealed package, one for the manufacturer, one for the purchaser, and one for an umpire, if necessary.

Section 6.—Revise to read as indicated above for Section 11 in Specifications B 139.

Table III.—In this table, for wire 0.025 in. and under, change the requirement for minimum tensile strength from the present "150,000" to read "145,000" psi. and delete the requirement of "3.5 per cent" for elongation tests on sizes over 0.125 to 0.250 in.

Section 8.—Revise to read as follows: "Grade A spring wire in sizes up to 0.250 in. in diameter, inclusive, shall withstand being bent cold through an angle of 120 deg. to a radius equal to the diameter of the wire and shall develop no cracks or flaws visible to the unaided eye."

Section 10.—Delete the note and revise the title to read "Dimensional Tolerances."

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[2] These revised specifications were accepted by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

Tentative Specifications for Aluminum Bronze Sheet and Strip (B 169 - 46 T):

Title.—Revise the title to read "Tentative Specifications for Aluminum Bronze Plate, Sheet, and Strip" and substitute "plate, sheet, and strip" for "sheet and strip" throughout the specifications.

Section 3.—Revise to read as indicated above for Section 3 in Specifications B 19.

Section 9.—Revise to read as indicated above for Section 11 in Specifications B 139.

Section 10.—Delete Note 2.

Sections 10, 11, and 13.—Combine in one section under the title "Dimensional Tolerances" and renumber subsequent sections accordingly.

Tentative Specifications for Copper Bus Bars, Rods, and Shapes (B 187 - 46 T):

Section 4.—Revise Paragraph (*a*) to read as indicated above for Section 3 (*a*) in Specifications B 19.

Section 10.—Retitle and revise Paragraph (*a*) to read as indicated above for Section 16 in Specifications B 133 omitting A.S.T.M. Designation: E 53.

Section 11.—Delete the note.

Sections 11 and 12.—Combine in one section under the title "Dimensional Tolerances" and renumber subsequent sections accordingly.

Section 13.—Revise to read as indicated above for Section 20 of Specifications B 133.

Table VII.—In Table VII, in the first column, delete the word "drawn" and also in the second line delete the words "bars and."

Tentative Specifications for Copper Bus Pipes and Tubes (B 188 - 46 T):

Section 4 (a).—Revise to read as indicated above for Section 3 (*a*) in Specifications B 75.

Section 10 (a).—Revise to read as indicated above for Section 12 in Specifications B 135, omitting A.S.T.M. Designation: E 36.

Section 11.—Delete the note.

Table III.—Begin the title and the heading of the first column with the word "Standard" and for type M, sizes up to and including 2-in. nominal, delete nominal wall thickness and tolerances and theoretical weight values.

Tentative Specifications for Beryllium-Copper Alloy Strip (B 194 - 46a T):

Title.—Revise the title to read "Tentative Specifications for Beryllium-Copper Alloy Plate, Sheet, and Strip" and substitute "plate, sheet, and strip" for "strip" throughout the specifications.

Section 3.—Reletter the present section as Paragraph (*b*) and add a new Paragraph (*a*) to read as indicated above for Section 3 (*a*) in Specifications B 19.

Section 4.—Revise to read as follows:

> 4. The material shall conform to the following requirements as to chemical composition:

Beryllium, per cent[a]	1.90 to 2.15
Additive elements:	
Nickel or cobalt, or both, min., per cent[a]	0.20
Nickel plus cobalt plus iron, max., per cent[a]	0.60[b]
Copper plus beryllium plus additive elements, min., per cent[a]	99.50[b]

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[a] For purposes of acceptance or rejection, an observed value or calculated value obtained from analysis should be rounded off to the nearest unit in the last righthand place of figures used in expressing the specified limit in accordance with the rounding off procedure prescribed in Section 4 of the Tentative Recommended Practices for Designation of Numerical Requirements in Standards (A.S.T.M. Designation: E 29).

[b] Iron in amounts less than 0.15 per cent is an unavoidable inclusion.

Section 5.—Revise to read as indicated above for Section 5 in Specifications B 159, except that in Paragraph (*c*) substitute the value of "100 g." for "150 g."

Table I.—Add a note to read as indicated above for Table I in Specifications B 36.

Section 11.—In the second line, insert the words "in case of disagreement," after the word "shall."

Section 12.—Delete Note 2.

Tables IV, V, and VI.—Add tolerances for sheet and plate as in the present Tables III, IV, and V of Specifications B 103.[3]

Sections 12, 13, and 15.—Combine in one section under the title "Dimensional Tolerances" and renumber subsequent sections accordingly.

Tentative Specifications for Beryllium-Copper Alloy Strip, Special Grade (B 195 - 46 T):

Sections 3, 4 and 5.—Revise to read as indicated above for Sections 3, 4, and 5 of Specifications B 194.

Section 11.—In the second line, insert the words "in case of disagreement" after the word "shall."

Section 12.—Delete the note.

Sections 12, 13, and 15.—Combine in one section under the heading "Dimensional Tolerances" and renumber subsequent sections accordingly.

Tentative Specifications for Beryllium-Copper Alloy Rod and Bar (B 196 - 46a T):

Section 3.—Reletter the present section as Paragraph (*b*) and add a new Paragraph (*a*) to read as indicated above for Section 3 (*a*) in Specifications B 19.

Section 4.—Revise to read as indicated above for Section 4 of Specifications B 194.

Section 5.—Revise Paragraph (*a*) to read as follows: "(*a*) Pieces from four individual rods or bars shall be taken for each lot of 5000 lb. or fraction thereof."

Add Paragraphs (*b*), (*c*), and (*d*) to read as indicated above for Section 5 in Specifications B 194.

Section 11.—In the second line add the words "in case of disagreement" after the word "shall."

Section 12.—Delete the note.

Section 14.—Revise to read as indicated above for Section 20 in Specifications B 133.

Tentative Specifications for Beryllium-Copper Alloy Wire (B 197 - 46a T:

Section 3.—Revise to read as follows: "The material shall be beryllium-copper alloy of such quality and purity that the finished product shall have the properties and characteristics prescribed in these specifications, and shall be finished by such cold drawing, annealing or heat treatment as may be required."

Section 4.—Revise to read as indicated above for Section 4 in Specifications B 194.

Section 5.—Revise to read as indicated above for Section 5 in Specifications B 194.

Section 7.—Revise the tensile strength requirements in Table II as follows, retaining the present footnotes *a* and *b* and deleting the present elongation values:

Condition	Tensile Strength, psi[a]	
	Revised Values	Present Values
A	58 000 to 78 000	80 000 max.
¼ H	90 000 to 115 000	90 000 min.
½ H	110 000 to 135 000	100 000 min.
¾ H[b]	130 000 to 155 000	125 000 to 145 000

New Section.—Add a new Section 9 to read as follows, renumbering the subsequent sections accordingly:

> 9. *Bending Properties.*—Wire as supplied commercially shall withstand being bent cold through an angle of 120 deg. to a radius equal to the diameter or thickness of the wire and shall develop no cracks or flaws visible to the unaided eye.

Section 9.—Revise to read as follows:

> At least four test specimens in any one shipment shall be taken from each lot of 5000 lb. or fraction thereof of the material of the same size in the solution-treated, cold-drawn, or rolled condition. Tension and bend tests shall be made on two specimens as supplied and tension tests only, on the remaining two specimens after precipitation hardening by heat treatment.

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[3] 1946 Book of A.S.T.M. Standards, Part I-B.

Section 11.—In the second line add the words "in case of disagreement" after the word "shall."

Section 12.—Delete the note.

Tentative Specifications for Copper-Nickel-Zinc Alloy Wire (B 206-46a T):

Section 1.—In Paragraph (*a*) add alloy B-1 having the following nominal composition:

Copper, per cent	60
Nickel, per cent	18
Zinc, per cent	22

In Paragraph (*b*) revise the heading "Alloy B" to read "Alloys B and B-1."

Section 3.—Revise to read as follows:

3. The material shall be copper-nickel-zinc alloy of such quality and purity that the finished product shall have the properties and characteristics prescribed in these specifications, shall be finished by the cold-drawing or cold-rolling and annealing processes, and may be furnished in either the cold-worked or annealed condition as specified.

Table I.—In Table I, add alloy B-1 with composition limits as follows:

Copper, per cent	58.5 to 61.5
Nickel, per cent	16.5 to 19.5
Lead, max., per cent	0.05
Iron, max., per cent	0.25
Manganese, max., per cent	0.50
Zinc, per cent	remainder

Add a footnote letter *b* after "Nickel, per cent" in column 3 and add footnote *b* to read "Cobalt counting as nickel."

For alloys A and B change the values for maximum lead content from the present "0.10" to read "0.05" per cent.

Section 6.—Revise to read as follows:

6. The tempers of drawn or rolled wire shall be designated as shown in Table II in the column titled "Cold-Worked Tempers" and for the various alloys and sizes are available only in the tempers shown.

NOTE.—Hexagonal, octagonal, rectangular and square wire are normally furnished only in the quarter-hard temper.

Section 7.—Revise to read as follows:

7. The tempers of annealed wire shall be designated as shown in Table III in the column titled "Nominal Grain Size" and for the various alloys are available only in the grain sizes shown.

Section 8.—Revise to read as follows:

8. The tension test shall be the standard test for all tempers of drawn or rolled wire and acceptance or rejection for temper shall be made on the basis of tensile strength only. Round wire 0.020 to 0.250 in., inclusive, in diameter shall conform to the requirements prescribed in Table II. The tensile strength values of wire smaller than 0.020 in., and larger than 0.250 in. in diameter shall be as agreed upon by the manufacturer and the purchaser. The tensile strength values for wire other than round shall likewise be subject to special agreement by the manufacturer and the purchaser.

Table II.—Revise to read as shown in the accompanying Table II, retaining the present footnote *a* and deleting footnotes *b* and *c*.

TABLE II.—TENSILE REQUIREMENTS FOR ROUND WIRE 0.020 TO 0.250 IN., INCLUSIVE, IN DIAMETER.

Temper	Tensile Strength, psi.[a]		
	Alloy *A*	Alloys *B* and *B*-1	Alloy *C*
Quarter hard	68 000 to 84 000	74 000 to 93 000	....
Half hard	83 000 to 97 000	92 000 to 110 000	75 000 to 95 000
Hard	99 000 to 111 000	112 000 to 128 0[illegible]	....
Spring:			
0.020 to 0.0253 in., incl.	....	130 000 min.	....
Over 0.0253 to 0.0625 in., incl.	....	125 000 min.	....
Over 0.0625 to 0.250 in., incl.	....	120 000 min.	....

Table III.—In Table III add a reference to alloy B-1 for each grain size.

New Section.—Add a new Section 10 to read as follows, renumbering the subsequent sections accordingly: "10. *Bending Properties.*—Spring temper wire in sizes up to 0.250 in., inclusive shall withstand being bent cold through an angle of 120 deg. to a radius equal to the diameter or thickness of the wire and shall develop no cracks or flaws visible to the unaided eye."

Section 10.—Revise the title to read "Number of Tests," and the first sentence to read as follows: "Bend tests whenever required, and tension tests shall be made on each lot of 5000 lb. or fraction thereof."

TABLE III.—THICKNESS TOLERANCES FOR RECTANGULAR AND SQUARE WIRE.

Thickness, in.	Width, in.	
	Up to ½, incl.	Over ½ to 1¼, incl.
	Tolerances, plus and minus[a], in	
Up to 0.050, incl.	0.0015	0.002
Over 0.050 to 0.090, incl.	0.002	0.003
Over 0.090 to 0.130, incl.	0.003	0.004
Over 0.130 to 0.187, incl.	0.004	0.0045

[a] When tolerances are specified as all plus or all minus, double the above values.

TABLE IV—WIDTH TOLERANCES FOR RECTANGULAR (NOT INCLUDING SQUARE) WIRE (FOR SQUARE WIRE USE THICKNESS TOLERANCES IN TABLE V (ACCOMPANYING TABLE III).

Width, in.	Tolerances, plus and minus[a], in.
Up to 0.050, incl.	0.0015
Over 0.050 to 0.090, incl.	0.002
Over 0.090 to 0.130, incl.	0.003
Over 0.130 to 0.187, incl.	0.004
Over 0.187 to 0.500, incl.	0.005
Over 0.500 to 1.25, incl.	0.007

[a] When tolerances are specified as all plus or all minus, double the above values.

Section 11.—Retitle and revise to read as indicated above for Section 13 (*a*) in Specifications B 19, listing only the following tests:

Test	A.S.T.M. Designation
Tension	E 8
Grain Size	E 2, E 3

Section 12.—Delete the note.

Tables V and VI.—Revise Tables V and VI to read as shown in the accompanying Tables III and IV, respectively.

Explanatory Note.—Add alloy B-1 with a density value of "0.315 lb. per cu. in."

Appendix.—Delete the Appendix.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends, for immediate adoption, revisions in 20 specifications as indicated below and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these modifications may be referred to letter ballot of the Society.

Standard Specifications for Copper Plates for Locomotive Fireboxes (B 11 – 46):

Section 3.—Designate the present section as Paragraph (*b*) and add a new Paragraph (*a*) to read as indicated above for Section 3 (*a*) in Specifications B 19.

New Section.—Add a new Section 10 reading as indicated above for Section 13 (*a*) in Specifications B 19, renumbering the subsequent sections accordingly, and listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 53, E 62
Tension	E 8

Standard Specifications for Copper Rods for Locomotive Staybolts (B 12 – 46):

Section 3.—Revise to read as follows:

3. (*a*) The material shall be of such quality and purity that the finished product shall have the properties and characteristics prescribed in these specifications.

(*b*) The rods shall be finished to dimensions by hot or cold working, or both, from suitable bars as required.

Section 11.—Retitle and revise to read as indicated above for Section 13 (*a*) in Specifications B 19, listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 53
Tension	E 8

Section 12.—Delete the note.

Standard Specifications for Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 - 46):

Section 3.—Reletter the present section as Paragraph (*b*) and add a new Paragraph (*a*) to read as indicated above for Section 3 (*a*) in Specifications B 19.

Table I.—Revise Table I to read as shown in the accompanying Table V.

Section 7.—Add a new Paragraph (*c*) to read as follows: "(*c*) Half-hard temper rods and bars shall be furnished unless otherwise specified in the contract or purchase order. Physical requirements of drawn shapes shall be agreed upon between the manufacturer and the purchaser at the time of placing the order.

TABLE V.—TENSILE REQUIREMENTS.

Temper	Nominal Diameter or Distance Between Parallel Faces, in.		Tensile Strength, min., psi.	Yield Strength, min., psi.	Elong. in 2 in., min., per cent	Rockwell B Hardness on Surface, min. (For information only)	
	ROUNDS, HEXAGONS AND OCTAGONS					ROUNDS	HEXAGONS OCTAGONS
Soft	1 and under		48 000	20 000	20	..	..
	Over 1 to 2 incl		44 000	18 000	25	..	..
	Over 2		40 000	15 000	30	..	..
Half Hard	½ and under		57 000	25 000	7	..	..
	Over ½ to 1 incl		55 000	25 000	15	65	60
	Over 1 to 2 incl		50 000	20 000	20	60	50
	Over 2		45 000	15 000	25	60	45
Hard	⅛ to 3/16 incl		80 000	45 000	..	..	..
	Over 3/16 to 5/16		70 000	35 000	4	..	..
	RECTANGLES AND SQUARES						
	Thickness, in.	Width, in.					
Soft	1 and under	6 and under	44 000	18 000	20	..	
	Over 1	6 and under	40 000	15 000	25	..	
Half Hard	½ and under	1 and under	50 000	25 000	10	60	
	½ and under	Over 1 to 6	45 000	17 000	15	50	
	Over ½ to 2	2 and under	45 000	17 000	20	50	
	Over ½ to 2	Over 2 to 6	40 000	15 000	20	40	
	Over 2	Over 2 to 4	40 000	15 000	20	40	

Section 11.—Revise to read as indicated above for Section 16 in Specifications B 133, except in the list of tests replace "E 53" with "E 36."

Section 12.—Delete the note.

Section 13.—Combine with Section 12 and renumber subsequent sections accordingly.

Section 14.—Revise to read as indicated above for Section 20 in Specifications B 133.

Standard Specifications for Copper Pipe, Standard Sizes (B 42 - 46):

Section 3.—Revise to read as indicated above for Section 3 (*a*) in Specifications B 75.

Section 4.—Renumber as Section 6 to follow present Section 6 and change title to read "Temper."

Section 7 (a).—Delete the last sentence.

New Section.—Add a new Section 11 to read as indicated above for Section 10 in Specifications B 11, renumbering the subsequent sections accordingly, and listing the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 53, E 62
Expansion (Pin Test)	B 153
Microscopic Examination	E 2, E 3

Section 13.—Delete the note.

Sections 13 and *14.*—Combine in one section under the title "Weight and Dimensional Tolerances" and renumber subsequent sections accordingly.

Section 14 (a).—Delete the word "recommended" from the first sentence.

Standard Specifications for Red Brass Pipe, Standard Sizes (B 43 - 46):

Section 3.—Revise to read as follows: "3. The material shall be copper-zinc alloy of such quality and purity that the finished product shall have the properties and characteristics prescribed in these specifications, and shall be cold drawn to size."

Section 4.—Renumber as Section 6 to follow present Section 6 and change the title to read "Temper."

Section 12.—Retitle and revise to read as indicated above for Section 13 (*a*) in Specifications B 19, listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 36
Expansion (Pin Test)	B 153
Mercurous Nitrate	B 154
Microscopic Examination	E 2, E 3

Section 14.—Delete the note.

Sections 14 and *15.*—Combine in one section under the title "Weight and Dimensional Tolerances" and renumber subsequent sections accordingly.

Section 15 (a).—Delete the word "recommended" in the first sentence.

Standard Specifications for Seamless Copper Tubing, Bright Annealed (B 68 - 46):

Section 1.—Add a new sentence to read as follows: "Three types are specified as follows:

Type A.—Phosphorized copper, low residual phosphorus.

Type B.—Phosphorized copper, high residual phosphorus.

Type D.—Oxygen-free copper without residual metallic deoxidants."

Section 3 (a).—Revise to read as indicated above for Section 3 (*a*) in Specifications B 75.

Section 4.—For type A, change the maximum permissible phosphorus content from the present "0.010" to read "0.012" per cent.

Section 12.—Retitle "Methods of Test and Chemical Analysis" and revise to read as indicated above for Section 13 (*a*) in Specifications B 19, listing the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 53, E 62
Tension	E 8
Grain Size	E 2, E 3
Expansion (Pin Test)	B 153

Section 13.—Delete the note.

Table I.—In Table I rearrange to read as indicated above for Table II in Specifications B 75.

Sections 13 and *14.*—Combine in one section under the title "Dimensional Tolerances" and renumber subsequent sections accordingly.

Standard Specifications for Copper Water Tube (B 88 - 46):

Table I.—Revise Table I to read as shown in the accompanying Table VI.

TABLE VI.—TENSILE STRENGTH AND GRAIN SIZE REQUIREMENTS.

Temper	Nominal Size, in.	Tensile Strength, min., psi.	Rockwell Hardness			Grain Size, mm.
			B	F	30-T	
Annealed	All	30 000	....	50 max.	....	0.040 min.
Drawn	All	36 000	20 min.	55 min.	30 min.	....

Section 12.—Retitle "Methods of Test and Chemical Analysis" and revise to read as follows:

12. (*a*) The properties and chemical compositions enumerated in these specifications shall, in case of disagreement, be determined in accordance with the following methods of the American Society for Testing Materials, with the modification given in Paragraph (*b*):

Test	A.S.T.M. Designation
Chemical Analysis	E 53, E 62
Tension	E 8
Rockwell Hardness	E 18
Grain Size	E 2, E 3
Expansion (Pin Test)	B 153

(*b*) *Grain Size.*—The test specimen for the microscopic examination shall be a radial longitudinal section of the tube made by cutting along a plane containing the longitudinal axis of the tube.

Section 15.—Delete the note.

Section 16.—Combine with Section 15, renumbering subsequent sections accordingly.

Standard Specifications for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 – 46):

Section 1.—Add the words "and annealed" after the word "rolled" in the first sentence.

Section 2.—Delete Item (*2*), renumbering subsequent items accordingly.

Section 3.—Designate the present Section as Paragraph (*b*) and revise to read: "The plate and sheet shall be manufactured by hot rolling and annealing or by cold rolling and annealing." Add a new Paragraph (*a*) to read as indicated above for Section 3 (*a*) in Specifications B 19.

Section 5.—Change the heading to read: "Sampling for Chemical Analysis" and revise Paragraphs (*a*) and (*b*) to read as follows:

5. (*a*) A sample shall be taken from each 5000 lb. or fraction thereof. The sample shall consist of equal quantities taken from three pieces. If the lot be of less than three pieces, a sample shall be taken from each piece.

(*b*) The samples for chemical analysis shall consist of drillings or millings taken from representative plates, and shall be so taken as to represent accurately material from the full thickness of the plate. The samples for chemical analysis may be taken from the specimens used for mechanical testing.

Add Paragraph (*c*) to read as follows:

(*c*) The minimum weight of sample required for analysis is 150 g. The drillings or millings shall be mixed thoroughly and shall be divided into three equal parts, one for the manufacturer, one for the purchaser and one for an umpire, if necessary.

Add Paragraph (*d*) to read as indicated above for Paragraph (*d*) in Specifications B 159.

Section 6.—Revise this section to read as follows:

6. The annealed plate and sheet shall conform to the following requirements as to tensile properties (values for Rockwell hardness and grain size are given for information only):

Tensile strength, psi	50 000 to 67 000
Yield strength, min., psi[a]	18 000
Elongation in 2 in., min., per cent	40
Rockwell Hardness, F scale[b]	72 to 92
Grain size, max., mm.[b]	0.110[c]

[a] Stress corresponding to 0.5 per cent strain.

[b] Hardness and grain size values given only as information.

[c] No minimum grain size is indicated, but all annealed material shall be fully recrystallized.

Table II.—Delete Table II, renumbering Table III as Table II.

Section 7.—Retitle and revise to read as indicated above for Section 13 (*a*) in Specifications B 19, listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 54
Tension	E 8[a]
Grain Size	E 2, E 3
Rockwell Hardness	E 18

[a] The tension test specimen shall conform to the dimensions shown in Figs. 2 or 3 of Standard Methods E 8.

Section 9. —Delete the note.

Standard Specifications for Copper-Silicon Alloy Sheet and Strip for General Purposes (B 97 – 46):

Title.—Revise the title to read "Standard Specifications for Copper-Silicon Alloy Plate, Sheet, and Strip for General Purposes" and substitute the words "plate, sheet, and strip" for "sheet and strip" throughout the specifications.

Section 2.—Add a new Item (*4*) to read as follows: "(*4*) Finish (Section 14 (*a*))."

Section 3.—Revise the title to read "Manufacture." Delete Paragraph (*b*), designate the present Paragraph (a) as (*b*), and revise to read: "The material shall be manufactured by hot rolling or cold rolling followed by such annealing as may be required." Add a new

Paragraph (*a*) to read as indicated above for Section 3 (*a*) in Specifications B 19.

Section 6.—Delete Paragraph (*b*).

Table II.—For types A and C add the following:

Temper	Tensile Strength, psi.
Hot rolled	55 000 to 72 000
Hot rolled, cold-rolled finish	58 000 to 72 000

Table III.—For types A and C add the following:

Temper	Rockwell Hardness F Scale	Rockwell Hardness B Scale
Hot rolled	72 min.	....
Hot rolled, cold-rolled finish	....	60 to 80

Combine Table III editorially with Table II, renumbering the subsequent tables accordingly, and add a footnote to be referenced after half-hard, hard, extra-hard, and spring tempers to read as follows: "Commercially supplied only as strip. The manufacturer should be consulted where these tempers are desired in sheet or plate."

Section 9.—Revise to read as indicated above for Section 7 in Specifications B 96.

Section 10.—Delete Note 2.

Sections 10, 11, and 13.—Combine in one section under the title "Dimensional Tolerances" and renumber subsequent sections accordingly.

New Section.—After Section 10, insert a new Section 11 titled "Weight Tolerances for Hot-Rolled Material" to read as follows, renumbering the subsequent sections accordingly:

11. The weight of each lot of five or more plates or sheets of the same specified dimensions when ordered to thickness shall not vary from the nominal by more than the amount prescribed in Table VI (accompanying Table VII). The weight of any individual plate or sheet may vary from the nominal by not more than one third in excess of the tolerance prescribed in Table VI (accompanying Table VII). The tolerances of lots of less than five plates or sheets shall be governed by the tolerance of individual plates or sheets. For purposes of these specifications, types A and C shall be considered to weigh 0.308 lb. per cu. in.

New Table.—Add a new Table VI to read as shown in the accompanying Table VII, renumbering the subsequent tables accordingly."

Section 14.—Retitle "Workmanship and Finish," renumber as Section 13, and revise to read as follows:

13. (*a*) Copper-silicon alloy plate and sheet are regularly supplied in the following finishes:

Black.—After hot rolling retains all of the oxides.

Plain Pickled.—Sulfuric acid pickle only. Brick red color. Has cuprous and silicon oxides still adherent.

Specially Cleaned.—Commercially free of all oxides. Has golden color of the alloy.

Sand Blasted.—Commercially free of all oxides. Has dull gray color.

(*b*) The plate and sheet shall be free from injurious defects of a nature that will interfere with the purpose for which it is intended. The fracture shall be uniform in color and grain throughout. Material shall be free from dirt and all sections, except black, shall be well cleaned.

Standard Specifications for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 – 46):

Section 3.—Reletter the present section as Paragraph (*b*) and add a new Paragraph (*a*) to read as indicated above for Section 3 (*a*) in Specifications B 19.

TABLE VII.—LOT OVERWEIGHT TOLERANCES OF PLATE AND SHEET ORDERED TO THICKNESS.

Thickness, in.	Permissible Excess in Average Weight of Lots, Expressed in Percentage of Nominal Weights					
	48 in. and Under in Width	Over 48 to 60 in. in Width	Over 60 to 72 in. in Width	Over 72 to 96 in. in Width	Over 96 to 120 in. in Width	Over 120 to 132 in., incl., in Width
1/8 to 3/16	6.5	8	9	11	..	..
Over 3/16 to 1/4	6.5	8	9	11	12	..
Over 1/4 to 5/16	6.5	7.75	8.75	11	12	13
Over 5/16 to 3/8	6.25	7.5	8.5	11	12	13
Over 3/8 to 7/16	6	7.25	8.25	11	12	13
Over 7/16 to 1/2	6	7	8	10	11	12
Over 1/2 to 5/8	5.75	6.5	7.5	9	10	11
Over 5/8 to 3/4	5.5	6	7	8	9	10
Over 3/4 to 1	5	5	6.25	7	8	9
Over 1 to 2	3.5	4	5	6	7	8

Section 10.—Revise to read as indicated above for Section 11 in Specifications B 139.

Section 11.—Delete the note.

Section 12.—Combine with Section 11 and renumber subsequent sections accordingly.

Section 13.—Add a note to read as indicated above for Section 20 of Specifications B 133.

Explanatory Note.—Revise to read as follows: "NOTE.—For purposes of these specifications, the density shall be taken as 0.308 lb. per cu. in. for types A, C, and D, and 0.316 lb. per cu. in. for type B."

Standard Specifications for Copper-Silicon Alloy Wire for General Purposes (B 99 – 46):

Table I.—Delete type D from this table.

Section 5.—Revise to read as indicated above for Section 5 in Specifications B 159.

Section 6.—Revise to read as follows:

> 6. (*a*) *Grain Size of Annealed Tempers.*—The average grain size of each of two samples of annealed wire as determined on the longitudinal section of the wire shall not exceed 0.040 mm. Although no minimum grain size is required, this material shall be fully recrystallized.
>
> (*b*) *Tensile Properties.*—The tensile properties of the material shall conform to the requirements prescribed in Table II.

Table II.—Revise Table II to read as shown in the accompanying Table VIII.

Section 7.—Revise to read as follows: "7. All wire up to and including $\frac{1}{4}$ in. in diameter shall withstand wrapping one full turn (360 deg.) around its own diameter and shall develop no cracks or flaws visible to the unaided eye. Bend tests shall not be made upon specimens which include brazes or welds."

Section 8.—Delete and renumber subsequent sections accordingly.

Section 10.—Revise to read as indicated above for Section 11 in Specifications B 206.

TABLE VIII.—TENSILE REQUIREMENTS OF COPPER-SILICON ALLOY WIRE.

Temper	Tensile Strength, psi.[a]	Elongation in 2 in., min. per cent, for wire over ¼ in. in Diameter
TYPES A AND C ALLOY WIRE		
Annealed	55 000 to 70 000	47
Eighth-hard	62 000 to 78 000	28
Quarter-hard	72 000 to 90 000	18
Half-hard	90 000 to 110 000	10
Hard[b]	115 000 to 135 000	6
Spring[c]	130 000 min.	4
TYPE B ALLOY WIRE		
Annealed	43 000 to 55 000	40
Eighth-hard	50 000 to 65 000	20
Quarter-hard	60 000 to 75 000	15
Half hard	75 000 to 95 000	10
Hard[b]	90 000 to 110 000	8
Spring[c]	100 000 min.	6

[a] For purposes of acceptance or rejection, an observed value or calculated value obtained from the tension test should be rounded off to the nearest 1000 psi. in accordance with the rounding off procedure prescribed in Section 4 of the Tentative Recommended Practices for Designation of Numerical Requirements in Standards (A.S.T.M. Designation: E 29).

[b] Hard temper is not generally available in sizes over ½ in.

[c] Spring temper is not generally available in sizes over ¼ in.

Section 11.—Delete the note.

Table III.—Delete type D from this table.

Explanatory Note.—Delete type D.

Table IV.—Delete type D from this table.

Standard Specifications for Rolled Copper-Alloy Bearing and Expansion Plates for Bridge and Other Structural Uses (B 100 – 46):

Title.—Revise the title to read "Standard Specifications for Rolled Copper-Alloy Bearing and Expansion Plates and Sheets for Bridge and Other Structural Uses" and substitute "plates and sheets" for "plates" throughout the specifications.

Section 1.—Revise the last sentence to read as follows: "A phosphor bronze alloy designated as type A and a copper-silicon alloy designated as type B are covered by these specifications."

Section 3 (a).—Revise to read as indicated above for Section 3 (*a*) in Specifications B 19.

Section 5.—Revise to read as follows:

5. (*a*) A sample shall be taken from each 2,000 lb. or fraction thereof. The sample shall consist of equal quantities taken from three pieces. If the lot be of less than three pieces, a sample shall be taken from each piece.

(*b*) The samples for chemical analysis shall consist of drillings or millings taken from the plates selected for purpose of tests as specified in Sections 6 and 7, and shall be so taken as to represent accurately material from the full thickness of the plate. The samples for chemical analysis may be taken from the specimens used for mechanical testing.

(*c*) The minimum weight of sample required for analysis is 100 g. for type A and 150 g. for type B. The drillings or millings shall be mixed thoroughly and shall be divided into three equal parts, one for the manufacturer, one for the purchaser, and one for an umpire, if necessary.

(*d*) In case of dissatisfaction, either party may require that the material be resampled in the presence of representatives of the manufacturer and the purchaser. The thoroughly mixed sample shall be divided into three equal parts, each of which shall be placed in a sealed package, one for the manufacturer, one for the purchaser, and one for an umpire, if necessary.

Section 7.—Retitle "Methods of Test and Chemical Analysis" and revise to read as follows:

7. (*a*) The properties and chemical compositions enumerated in these specifications shall, in case of disagreement, be determined in accordance with the following methods of the American Society for Testing Materials, with the modification given in Paragraph (*b*):

Test	A.S.T.M. Designation
Chemical Analysis	E 54
Tension	E 8
Rockwell Hardness	E 18
Brinell Hardness	E 10

(*b*) *Compression Tests.*—Compression test specimens shall be 1 sq. in. in cross-sectional area and the height shall be the thickness of the plate from which the specimen is taken. The yield strength in compression shall be determined by successive applications and releases of the load until a 0.1 per cent offset is determined.

Section 8.—Delete the note.

Standard Specifications for Phosphor Bronze Sheet and Strip (B 103 – 46):

Title.—Revise the title to read "Standard Specifications for Phosphor Bronze Plate, Sheet, and Strip" and substitute "plate, sheet, and strip" for "sheet and strip" throughout the specifications.

Section 3.—Revise Section 3 to read as indicated above for Section 3 in Specifications B 19.

Section 6.—Delete and renumber subsequent sections accordingly.

Table II.—Delete the words "Temper and" from the title. Delete the columns headed "Numbers Hard" and "Nominal Reduction by Rolling, per cent." Add a note to the table to read as indicated above for Table I in Specifications B 36.

Section 12.—Retitle and revise to read as indicated above for Section 13 (*a*) in Specifications B 19, listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 54
Tension	E 8
Rockwell Hardness	E 18

Section 13.—Delete Note 2.

Sections 13, 14, and 16.—Combine in one section under the title "Dimensional Tolerances" and renumber subsequent sections accordingly.

Standard Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 46):

Section 3.—Retitle "Manufacture" and revise to read as follows: "3. The material shall be copper or copper alloy as specified, of such quality and purity that the finished product shall have the properties and characteristics prescribed in these specifications, and shall be cold worked to the specified size."

Section 4.—Retitle "Temper" and delete Paragraph (*a*).

Section 5.—In Table I, in the first column, after "80-20 copper-nickel" alloy, add "90-10 copper-nickel" alloy with the following chemical composition:

Copper, per cent	remainder
Nickel, per cent	9.0 to 11.0
Lead, per cent	0.05 max.
Iron, per cent	1.0 max.
Zinc, per cent	1.0 max.
Manganese, per cent	1.0 max.

Section 7 (a).—Revise to read as follows:

7. (*a*) Samples of annealed-temper tubes selected for test shall be subjected to microscopic examination at a magnification of 75 diameters and shall show uniform and complete recrystallization. Alloys other than Muntz metal shall have an average grain size within the limits of 0.010 to 0.045 mm. The requirements of Section 7 do not apply to tubes of light drawn, hard drawn or hard drawn end annealed tempers.

Section 7.—Delete Paragraph (*c*).

Section 8.—Delete Paragraph (*c*).

Section 10.—Revise the last sentence to read as follows: "The mercurous nitrate test is not required for copper, arsenical-copper or copper-nickel tubes."

New Section.—Add a new Section 12 to read as indicated above for the new Section 10 in Specifications B 11, renumbering the subsequent sections accordingly, and listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 36, E 53, E 54, E 62
Grain Size	E 2, E 3
Expansion (Pin Test)	B 153
Mercurous Nitrate	B 154

Section 14 (c).—Delete the note.

Section 14 (d).—After the alloy "80-20 copper-nickel," add the alloy "90-10 copper-nickel" with a unit weight of "0.323 lb. per cu. in."

Standard Specifications for Copper and Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 46):

Section 3.—Revise to read as indicated above for Section 3 in Specifications B 19.

Table I.—In the table, for alloy No. 3, revise the copper content from the present "59 to 61" to read "59.0 to 62.0" per cent.

Section 6.—Delete the note.

New Section.—Add a new Section 6 to read as follows and renumber subsequent sections accordingly:

6. *Methods of Chemical Analysis.*—The chemical compositions enumerated in these specifications shall, in case of disagreement, be determined in accordance with the following methods of the American Society for Testing materials:

Test	A.S.T.M. Designation
Chemical Analysis	E 36, E 53, E 54

Section 7.—Combine with the present Section 6 and renumber subsequent sections accordingly.

Standard Specifications for Brass Wire (B 134 – 46):

Section 3.—Revise to read as follows: "3. The material shall be copper-zinc alloy of such quality and purity that the finished product shall have the properties and characteristics prescribed in these specifications, shall be finished by the cold-drawing or cold-rolling and annealing processes and may be furnished in either the cold-worked or annealed condition as specified."

Section 6.—Delete the columns headed "Nominal Reduction B. & S. Gage No." and "Approximate Percentage Reduction."

Section 8.—Revise to read as follows: "8. The tension test shall be standard for all tempers of drawn or rolled wire and acceptance or rejection shall depend only on the tensile strength. The tensile strength requirements specified in Table II shall determine acceptability of wire in all sizes 0.020 in. and over in diameter or thickness excepting, however, that these requirements shall not apply to hard, extra-hard, and spring temper wire in any sizes beyond the limits of availability defined in Table II for these respective tempers."

Table II.—Revise the title to read "Tensile Strength Requirements for Wire 0.020 in. and Over in Diameter or Thickness."

For alloy No. 4, spring temper, change the minimum tensile strength requirement of "120,000" to read "116,000" psi. Delete all values for alloy No. 6 and include a reference to alloy No. 6 in the column heading for alloy Nos. 7 and 8. For alloy Nos. 7 and 8, spring temper, change the minimum tensile strength requirement of "123,000" to read "120,000" psi.

Add a footnote letter *b* after "Hard" temper, the footnote to read "Hard temper wire is not generally available in sizes over ½ in. in diameter."

Add a footnote letter *c* after "Extra-Hard" temper, the footnote to read "Extra-hard temper is not generally available in sizes over ⅜ in. in diameter."

Add a footnote letter *d* after "Spring" temper, the footnote to read "Spring" temper is not generally available in sizes over ¼ in. in diameter."

Add a footnote letter *e* after "Extra-Hard" and "Spring" tempers, the footnote to read "Square and rectangular wire is not generally available in extra-hard or spring tempers."

Section 11.—Retitle and revise to read as indicated above for Section 13 (*a*) in Specifications B 19, listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis	E 36
Tension	E 8
Grain Size	E 2, E 3

Section 12.—Delete the note.

Tables V and VI.—Revise Tables V and VI to read as shown in the accompanying Tables IX and X, respectively.

Standard Specifications for Manganese Bronze Rods, Bars, and Shapes (B 138 – 46):

Section 3.—Reletter the present section as Paragraph (*b*) and add a new Paragraph (*a*) to read as indicated above for Section 3 (*a*) in Specifications B 19.

TABLE IX.—THICKNESS TOLERANCES FOR RECTANGULAR AND SQUARE WIRE.

Thickness, in.	Width, in.	
	Up to ½, incl.	Over ½ to 1¼, incl.
	Tolerances, plus and minus[a], in.	
Up to 0.013, incl.	0.001	0.0013
Over 0.013 to 0.050, incl.	0.0013	0.0015
Over 0.050 to 0.090, incl.	0.0015	0.002
Over 0.090 to 0.130, incl.	0.002	0.0025
Over 0.130 to 0.187, incl.	0.003	0.0035

[a] When tolerances are specified as all plus or all minus, double the above values.

TABLE X.—WIDTH TOLERANCES FOR RECTANGULAR (NOT INCLUDING SQUARE) WIRE (FOR SQUARE WIRE USE THICKNESS TOLERANCES IN TABLE V (SEE ACCOMPANYING TABLE IX.)).

Width, in.	Tolerances, plus and minus[a], in.
Up to 0.050, incl.	0.0013
Over 0.050 to 0.090, incl.	0.0015
Over 0.090 to 0.130, incl.	0.002
Over 0.130 to 0.187, incl.	0.003
Over 0.187 to 0.500, incl.	0.0035
Over 0.500 to 1.25, incl.	0.005

[a] When tolerances are specified as all plus or all minus, double the above values.

Section 11.—Revise to read as indicated above for Section 11 in Specifications B 21, listing "E 54" in place of "E 36."

Section 12.—Delete the note.

Section 13.—Combine with Section 12 and renumber subsequent sections accordingly.

Section 14.—Revise to read as indicated above for Section 14 in Specifications B 21.

Standard Specifications for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 – 46):

Section 3.—Reletter the present section as Paragraph (*b*) and add a new Paragraph (*a*) to read as indicated above for Section 3 (*a*) in Specifications B 19.

Section 9.—Delete the note.

Section 10.—Combine with Section 9

and renumber subsequent sections accordingly.

New Section.—Add a new Section 9 to read as indicated above for the new Section 10 in Specifications B 11, renumbering the subsequent sections accordingly, and listing only the following tests:

Test	A.S.T.M. Designation
Chemical Analysis..............	E 36, E 54
Tension........................	E 8

Section 12.—Revise to read as indicated above for Section 20 in Specifications B 133.

Standard Method of Test for Expansion (Pin Test) of Copper and Copper-Alloy Tubing (B 153 – 45):

Section 1.—Add the following as a note:

NOTE.—For tubes of sizes greater than 4 in. in outside diameter the following flattening test, as described in the various pipe and tube specifications, is recommended as a substitute for the pin expansion test: For pipe and tubes over 4 in. in outside diameter in the annealed condition, a section 4 in. long shall be cut from the end of one of the tubes. This 4-in. long sample shall be flattened so that a gage set at three times the wall thickness will pass over the pipe freely throughout the flattened part. The pipe or tube so tested shall develop no cracks or flaws visible to the unaided eye as a result of this test. In making the flattening test, the elements shall be slowly flattened by one stroke of the press.

Standard Specifications for Copper-Alloy Condenser Tube Plates (B 171 – 46):

Section 3.—Revise to read as indicated above for Section 3 (*a*) in Specifications B 19.

Section 5.—Revise to read as indicated above for Section 5 in Specifications B 96.

Section 8.—Retitle "Methods of Test and Chemical Analysis" and revise Paragraph (*a*) to read as follows:

(*a*) The properties, except bending properties, and chemical compositions enumerated in these specifications shall, in case of disagreement, be determined in accordance with the following methods of the American Society for Testing materials:

Test	A.S.T.M. Designation
Chemical Analysis..............	E 36, E 54
Tension........................	E 8[a]

---

[a] The tension test specimen shall conform to the dimensions shown in Fig. 2 or Fig. 3 of Standard Methods E 8.

Section 9.—Delete the note.

Sections 9 and 10.—Combine in one section under the title "Dimensional Tolerances" and renumber subsequent sections accordingly.

REPORT OF COMMITTEE B-6[*]
ON
DIE-CAST METALS AND ALLOYS

Committee B-6 on Die-Cast Metals and Alloys held two meetings during the year: one in Buffalo, N. Y., on June 27, 1946, and one in Philadelphia, Pa., on February 25, 1947, with an attendance of 24 and 39, respectively.

A balance of $2525.68 was reported by Society Headquarters in the funds of Committee B-6 as of December 30, 1946.

At the present time, the committee consists of 81 members, of whom 39 are classified as producers, 21 as consumers, and 21 as general interest.

I. Revision of Tentatives

The committee recommends the following revisions in two tentative specifications:

Tentative Specifications for Magnesium-Base Alloy Die Castings (**B 94–44T**):[1]

Section 3.—Published corrosion data indicate that an increase in the copper content of alloy AZ91 will not adversely affect the corrosion resistance, and experience with even greater additions showed no harmful effect on properties, castability or serviceability of die castings. It is accordingly recommended that the copper requirement for alloy AZ91 be changed from "0.05 max." to "0.10 max." per cent.

Note 2.—Add the following to the end of this note: "in accordance with the rounding-off procedure prescribed in Section 4 of the Tentative Recommended Practices for Designation of Numerical Requirements in Standards (A.S.T.M. Designation: E 29).

Section 4.—Change this section to read as follows in accordance with the recommendation of Committee E-3 on Chemical Analysis of Metals:

4. *Sampling* (*a*) The sample for chemical analysis may be taken either by sawing, drilling, or milling a representative group of castings or test specimens, and shall represent the average cross-section of the pieces.

(*b*) The castings and the saw, drill, cutter, or other tool used for sampling shall be cleaned thoroughly. No lubricant shall be used in the sampling operation. Due care shall be exercised so that inserts, if present in the castings, will not contaminate the sample. The sawings or the metal chips shall be treated carefully with a strong magnet to remove any particles of iron introduced in taking the sample.

(*c*) The sample, the weight of which shall be agreed upon between the manufacturer and the purchaser, shall be divided into three equal parts, each of which shall be placed in a sealed package, one for the manufacturer, one for the purchaser, and one for an umpire, if necessary.

(*d*) In case of dissatisfaction, either party may require that the material be resampled in the presence of representatives of the manufacturer and the purchaser.

Section.—Add a new Section 5 to read as follows:

5. *Chemical Analysis.*—The chemical analysis shall be made in accordance with Standard Methods of Chemical Analysis of Magnesium and Magnesium-Base Alloys (A.S.T.M. Designation: E 35) or the Tentative Photometric Methods for Chemical Analysis of Magnesium and Magnesium-Base Alloys (A.S.T.M. Designation: E 61), or by any other approved methods agreed upon by the manufacturer and the purchaser. The analysis may be made spectrographically, provided that, in case of dispute,

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] 1946 Book of A.S.T.M. Standards, Part I-B.

the results secured by Method E 35 shall be the basis for acceptance.

Tentative Specifications for Copper-Base (Brass) Alloy Die Castings (**B 176–42 T**):[1]

Section 4.—Make the same change as recommended above for Section 4 of Tentative Specifications B 94.

Table I.—Add the following as a new footnote *a* and reletter the present footnote *a* as *b*:

"For purposes of acceptance and rejection, the observed value or calculated value obtained from analysis should be rounded off to the nearest unit in the last right-hand place of figures, used in expressing the specified limit in accordance with the rounding-off procedure prescribed in Section 4 of the Tentative Recommended Practices for Designation of Numerical Requirements in Standards (A.S.T.M. Designation: E 29)."

Section 5.—At the end of the note in this section, add the following paragraph: "By agreement between the manufacturer and the purchaser alloy A may be furnished with a higher elongation than the 10 per cent minimum."

II. TENTATIVE SPECIFICATIONS CONTINUED AS TENTATIVE

The committee recommends that the Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 – 46 T) be continued as tentative without revision.

The recommendations appearing in this report have been submitted to letter ballot[2] of the committee.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee IV on Physical Tests on Die Castings (R. L. Templin, chairman) offers the following tentative statement for further consideration by the membership and by Committee E-1 on Methods of Testing:

> In the tension testing of die-cast aluminum, magnesium, zinc, and brass alloy specimens, the rate of stressing up to the yield strength shall not exceed 100,000 psi. per minute. Exceedingly slow rates of testing may result in creep and hence are to be avoided. Beyond the yield strength, the rate of straining shall not exceed 0.25 in. per inch per minute measured on the gage length of the specimen.

Die-cast zinc, lead, and tin alloys are considered in a different category since they are not ordinarily tested for yield strength. The following tentative statement was suggested to cover these materials:

> In the tension testing of die-cast lead and tin alloy specimens, the rate of strain shall be ⅛ in. per inch per minute measured on the gage length of the specimen.

Subcommittee VII on Magnesium-Base Die-Casting Alloys (H. W. Schmidt, chairman) recommended that the Tentative Specifications for Magnesium-Base Alloy Die Castings (B 94 – 44 T) be revised and continued as tentative as covered earlier in this report.

Subcommittee VIII on Brass Die-Casting Alloys (J. C. Fox, chairman) recommended that the Tentative Specifications for Copper-Base (Brass) Alloy Die Castings (B 176 – 42 T) be revised as previously noted and continued as tentative.

This subcommittee also has under consideration the adoption of a third alloy, to be known as alloy C, in Specifications B 176 – 42 T. However, no final action will be taken on this 82 per cent copper, 4 per cent silicon brass alloy until mechanical and physical property data have been determined on test bars of this alloy.

Subcommittee VIII has also decided to determine certain mechanical and physical properties, such as compressive and shear strengths, thermal and electrical conductivity, etc., for the alloys covered in Specifications B 176.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Special Research Committee.—This group has been active during the past year in planning studies to determine the effects of die casting variables. It is now ready to undertake active work inasmuch as a standard die has been agreed upon for its co-operative tests. This group will be reorganized as Subcommittee IX on Die-Casting Processes.

This report has been submitted to letter ballot of the committee, which consists of 81 members; 72 members returned their ballots, of whom 67 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

G. L. WERLEY,
Secretary.

REPORT OF COMMITTEE B-7*
ON
LIGHT METALS AND ALLOYS, CAST AND WROUGHT

Committee B-7 held a meeting at the Annual Meeting of the Society on June 26, 1946. At this meeting R. A. Quadt, D. L. Colwell and R. B. Smith were elected chairmen of Subcommittees I, II, and VII, respectively.

During the year ten members were added to the committee and there were six removals. At the present time the committee consists of 94 members of whom 68 are voting members; 31 are classified as producers, 25 as consumers, and 12 as general interest members.

The committee is planning an extensive program of corrosion tests with Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys. Questionnaires with regard to this program have been circulated to both committees and have been summarized in order to determine the type of tests and the locations which are considered most desirable by the committee members. This program was discussed at a meeting of the Advisory Committee during the Spring Group Meetings at Philadelphia and will be submitted to the committee as a whole at the time of the annual meeting.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1946 Annual Meeting, Committee B-7 presented to the Society through the Administrative Committee on Standards, a number of recommendations. These included the following two new tentative specifications accepted September 9, 1946, and November 6, 1946, respectively:

Tentative Specifications for:

Magnesium-Base Alloy Extruded Round Tubing (B 217 - 46 T), and
Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings and Permanent Mold Castings (B 179 - 46 T).

Acceptance of Specifications B 179 - 46 T carried with it the withdrawal of the following:

Tentative Specifications for:

Aluminum-Base Alloys in Ingot Form for Sand Castings (B 58),
Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings (B 112), and
Aluminum-Base Alloys in Ingot Form for Die Castings (B 125).

Revisions in the following tentative specifications were also accepted by the Administrative Committee on Standards on November 6, 1946:

Tentative Specifications for:

Aluminum and Aluminum-Alloy Sheets and Plates (B 209 - 46 T),
Magnesium-Base Alloy Sheet (B 90 - 46 T),
Aluminum for use in iron and steel manufacture (B 37 - 46 T),
Aluminum-Base Alloy Sand Castings (B 26 - 46 T), and
Aluminum-Base Alloy Permanent Mold Castings (B 108 - 46 T).

All of the above new tentatives and revisions appear in the 1946 Book of A.S.T.M. Standards, Part I-B.

I. NEW TENTATIVE

The committee recommends for publication as tentative the proposed Specifications for Aluminum and Alumi-

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*** Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.**

num-Alloy Extruded Bars, Rods, and Shapes, as appended hereto.[1]

II. REVISION OF TENTATIVES

The committee is also recommending revision of several tentative specifications as follows:

Tentative Specifications for Magnesium-Base Alloy Sand Castings (**B 80 – 45 T**)[2]. —In Table I it is recommended that the copper limit for alloys AZ92 and AZ63 be changed from "0.05" to read "0.25" per cent maximum, and for alloys A8, A10, A12, AZ101 and M1 that it be changed from "0.05" to read "0.10" per cent maximum. It is also recommended that the **X** grade of casting alloys be removed from these specifications since experience has indicated these alloys are not commercially available.

Tentative Specifications for Magnesium-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (**B 93 – 45 T**)[2]. In Table I it is recommended that the copper limit be changed from "0.04" to read "0.20" per cent maximum for alloys AZ63 and AZ92 and from "0.04" to read "0.08" per cent maximum for alloys A8, A10, A12, AS100, AZ91, AZ101 and M1, and that the **X** grade alloys be deleted as in Specifications B 80.

Tentative Specification for Aluminum Sheet and Plate for Use in Pressure Vessels (**B 178 – 46 T**)[2].—These specifications have been revised to add aluminum alloys including the aluminum-manganese alloy formerly covered by Tentative Specification B 126. The revised specifications are appended hereto.[3]

Tentative Specification for Magnesium-Base Alloy Permanent Mold Castings (**B 199 – 45 T**)[2].—In Table I it is recommended that the copper limit be changed from "0.05" to read "0.25" per cent maximum for alloy AZ92 and from "0.05" to read "0.10" per cent maximum for alloy A10.

Tentative Specification for Aluminum and Aluminum Alloy Bars, Rods and Wire (**B 211 – 46 T**)[2].—The following revisions are recommended:

Section 1.—Change the second sentence to read as follows: "Cold-finished and rolled materials are covered (Note 2) and the class desired must be specified in the order."

Add a note to this section to read as follows: "NOTE.—While extruded products may be furnished to these specifications as an alternate for cold finished or rolled material, when they are specifically desired they should be ordered in accordance with the Tentative Specifications for Aluminum and Aluminum-Alloy Extruded Bars, Rods, and Shapes (A.S.T.M. Designation: B221)."

Section 4.—Change to read as follows:

> 4. Samples for chemical analysis shall be taken in accordance with the Tentative Method of Sampling Wrought Non-Ferrous Metals and Alloys for Determination of Chemical Composition (A.S.T.M. Designation: E 55), except that the weight of the prepared sample may be a minimum of 75 g. A portion shall be taken to represent each 2000 pounds or fraction thereof of a given type alloy in the shipment and a sample prepared from each portion. If agreed upon by the manufacturer and the purchaser, a composite sample representing all portions taken from the shipment may be used for the analysis in lieu of analyzing samples from each portion. If the manufacturer has made an analysis of the material during the course of manufacture, he shall not be required to sample and analyze the finished product.

Table I.—Change the aluminum content for alloy A2 from "remainder" to read "99.0 min." per cent. Delete the minimum manganese limit for alloy ZG42.

Note 3.—Add the following to this note: "in accordance with Section 4 of Recommended Practices E 29."

Table II.—Change the minimum

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[1] These specifications were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

[2] 1946 Book of A.S.T.M. Standards, Part I-B.

[3] These revised specifications were accepted by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

elongation for alloy ZG42 annealed material 0.125 in. and over from "6 per cent" to read "10 per cent," and for heat-treated material 0.125 in. and over from "6 per cent" to read "7 per cent."

Table V.—Change the least distance between parallel faces, from "2.001 to 3.000" to read "2.001 to 4.000" in.

Table VI.—Delete this table and omit reference to it in Section 11.

III. WITHDRAWAL OF TENTATIVE SPECIFICATIONS

Tentative Specifications for Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (**B 126 – 46 T**).[2]—As the revision of Tentative Specifications B 178 – 46 T, referred to above, includes the requirements covered by these specifications, the withdrawal of Specifications B 126 – 46 T is recommended.

IV. TENTATIVE SPECIFICATIONS CONTINUED AS TENTATIVE

All the remaining tentative specifications under the jurisdiction of the committee, including the Tentative Specifications for Magnesium-Base Alloy Forgings B 91 – 45 T, are recommended for continuation as tentative without revision.

The recommendations appearing in this report have been submitted to letter ballot[4] of the committee, the results of which will be reported at the Annual Meeting.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum and Aluminum-Alloy Ingots (R. A. Quadt, chairman) completely revised the specifications under its jurisdiction last year. For this reason it has been unnecessary for the subcommittee to hold any meetings and further work will be contingent on the activities of Subcommittee II.

Subcommittee II on Aluminum Alloy Castings (D. L. Colwell, chairman).—An investigation of aluminum alloys for plaster mold casting and for centrifugal casting is being initiated, but before specifications are prepared for these alloys, a study of their present status will be reported to the committee.

Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys (P. V. Faragher, chairman) prepared the new specifications for aluminum alloy extrusions; the revisions in Specifications B 178 (including withdrawal of B 126); and the revisions in B 211, already mentioned.

Subcommittee IV on Magnesium and Magnesium Alloys, Cast and Wrought (H. W. Schmidt, chairman).—This subcommittee has reviewed data as to the effect of copper in the magnesium alloys. This led to the recommended changes in copper limits in Specifications B 80, B 93, and B 199, mentioned above. A number of changes in Specification B 107 – 45 T were discussed. It is expected that revision of these specifications will be submitted to the subcommittee for letter ballot in the near future.[5]

Subcommittee V on Testing Light Metals (R. L. Templin, chairman) held two meetings during the year at which attention was given to speed of testing as it applies to the tension testing of light alloys. After much discussion and further consideration by correspondence the subcommittee recommended that the following statement be published in its report so that it could be studied by those interested:

> The values obtained for the mechanical properties covered by these specifications are not seriously affected by variations in speed of

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[4] **The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.**

[5] **See Editorial Note, p. 185.**

testing. A considerable range of testing speed is permissible; however, the rate of stressing to the yield strength should not exceed 100,000 psi. per min., and above the yield strength the movement per minute of the head under load should not exceed 0.5 in. per in. of gage length (or distance between grips for specimens not having reduced sections). Care must be exercised, especially when making yield strength determinations, that the speed of testing should not exceed the ability of the strain and load-indicating equipment to function satisfactorily.

This statement will be considered for inclusion in all specifications under the jurisdiction of Committee B-7 that require tension tests. The subcommittee will be pleased to receive comments on this matter from those interested.

The tension testing of thin-walled magnesium tubing is being studied as is also the use and misuse of hardness in connection with light alloys.

A paper on "The Determination of Elongation of Sand-Cast Light Alloy Test Bars," by F. M. Howell was presented at a meeting of this subcommittee in Philadelphia at the time of the Spring Group Meetings. This paper appears as an Appendix to this report.

Subcommittee VI on Anodic Oxidation of Aluminum and Aluminum Alloys (J. D. Edwards, chairman).—The thickness of an oxide coating may be an important factor in determining the quality of the coating for various applications. In 1946, there became available to the public an electronic thickness gage which seemed admirably adapted to the nondestructive measurement of anodic coatings on aluminum. A comprehensive investigation of the performance of this instrument on various aluminum alloys has been made at Aluminum Research Laboratories. This investigation has shown that, when certain requirements in its use are fulfilled, the instrument is capable of giving precise measurements of thickness on oxide-coated wrought aluminum alloys. The techniques of making these tests and a discussion of the advantages and limitations of the instrument will be presented in a report now under preparation for publication.

Subcommittee VII on Codification of Light Alloys (R. B. Smith, chairman).—Two minor modifications of the code system for aluminum alloys were proposed at the last meeting of this subcommittee. These were submitted to a letter ballot of the committee, but were rejected in favor of retention of the present code.[6] However, there is evidence that improvement of the system is desired, so other modifications will be considered when suggested. In addition, the subcommittee is attempting to develop a system of temper designations for both aluminum and magnesium alloys.

This report has been submitted to letter ballot of the committee, which consists of 68 voting members; 48 members returned their ballots, of whom 46 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

I. V. WILLIAMS,
Chairman.

J. J. BOWMAN,
Secretary.

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[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 139 (1945).

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Aluminum and Aluminum Alloy Bars, Rods and Wire (B 211 - 46 T), and
Magnesium Base Alloy Bars, Rods and Shapes (B 107 - 45 T).

These recommendations were accepted by the Standards Committee on September 4, 1947, and the revised tentatives appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

On October 31, 1947, the Standards Committee accepted the following recommendations:

Revision of Tentative Specifications for:

Aluminum-Base Alloy Sand Castings (B 26 - 46 T),
Aluminum-Base Alloy Permanent Mold Castings (B 108 - 46 T), and
Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 - 46 T).

The revised specifications also appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

APPENDIX

THE DETERMINATION OF ELONGATION OF SAND-CAST LIGHT ALLOY TEST BARS*

By F. M. Howell[1]

There are a number of specifications for cast light alloys in which the requirements for elongation in 2 in. are specified to 0.5 per cent. These alloys have low elongations (1 to 5 per cent), and yet experience has shown that they are very useful. According to the Standard Methods of Tension Testing of Metallic Materials (E 8 – 46),[2] elongation in 2 in. is measured only to the nearest 0.5 per cent. For the materials under consideration differences of 0.5 per cent in 2 in. in measuring the elongation may affect the result by as much as 50 per cent of the value specified. This may lead to controversies as to whether a given lot of castings represented by the test bars complies with the requirements of the specification involved. The question of the practicability of determining elongation in 2 in. within closer limits is thus presented.

Purpose of Investigation

In order to obtain some factual information on the minimum limits within which it might reasonably be expected that elongation values could be determined, an investigation involving the testing of some 650 sand-cast aluminum alloy test bars was undertaken. Cognizance was taken of a number of factors that can affect elongation determinations. These have to do with the dimensions of the bars, the methods of gripping the bars during testing, the marking of the original gage length, and the measurement of the gage length after testing.

Preparation of Specimens

For this work two alloys were selected, alloy CN21 (Alcoa No. 142-T75) and alloy C1 (Alcoa No. 195-T6). About 400 bars of alloy CN21 and 250 bars of alloy C1 were cast in accordance with standard foundry practice. The bars were standard in all respects, but some were cast with shanks 3 in. long and some with the usual 2-in. length of shank. They were cast in 2-bar molds and each bar was identified so as to show in which side of the mold it was cast. Radiographs were made of all bars and they were all found to be commercially sound. The bars were also carefully inspected for straightness. The bars of alloy CN21 were from two different melts, but those of alloy C1 were all from the same melt. All bars from a given melt were heat treated together.

The minimum cross-sectional area of each bar was determined by making several diameter measurements with micrometer calipers. The double-pointed gage marker used for applying the gage marks to the specimens was carefully checked. The points were quite sharp with a radius of about 0.001 in. on each point; the distance between the two points was within 0.002 in. of 2 in. The gage marker was rechecked

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Chief, Mechanical Testing Div., Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[2] 1946 Book of A.S.T.M. Standards, Part I-A, p. 698; Part I-B, p. 296.

after marking 50 specimens and was found to be unchanged.

METHOD OF TEST

Tension Tests:

Tension tests were made in an Amsler universal testing machine having a maximum capacity of 20,000 lb. using the 10,000-lb. load range. Specimens with straight cylindrical ends were held in self-aligning Templin grips with V-wedges designed for gripping round specimens having $\frac{3}{4}$-in. diameter ends. These grips were thoroughly cleaned and lubricated at intervals during the test program. Specimens with threaded ends were held in threaded adapters. Both the Templin grips and the threaded adapters were attached to tension bolts that had spherical seats at their extreme ends. In the tests of alloy CN21 no attempt was made to determine yield strength because of the extremely low plastic deformation, but in the tests of alloy C1, yield strengths were determined by means of autographic load-strain curves. The speed of testing was held reasonably constant by setting the control valve at a reading that corresponds to a free-running speed of pulling head of the testing machine of 0.2 in. per minute.

Elongation:

Elongation in 2 in. was measured using dividers and scale. The two halves of each specimen were matched and then held together in the device shown in Fig. 1. This device applies a uniform pressure of about 20 lb. to the ends of the specimens by means of the spring-loaded spindle at the left side of the photograph. The support for the other spindle is adjustable, thus permitting the device to hold specimens ranging in total length from 2 to 9 in. The pressure

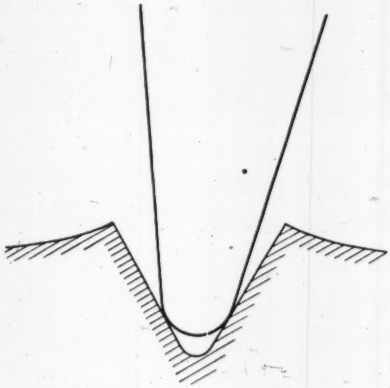
Radius at bottom of gage mark 0.001 in.
Radius on end of dividers point 0.003 in.

Fig. 2.—Enlarged Sketch Showing Seating of Point of Dividers in Gage Mark.

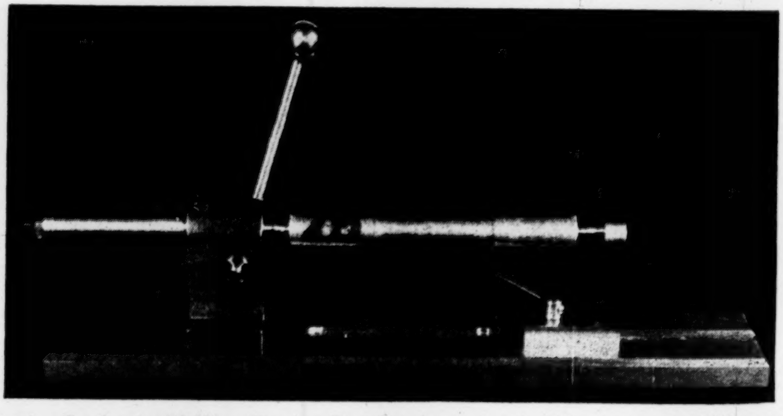

Fig. 1.—Device for Holding Tension Test Specimens for the Measurement of Elongation.

applied by the spring can be adjusted as desired up to a maximum of about 40 lb. A pressure of about 20 to 30 lb. has been found satisfactory.

The points of the 6-in. dividers were rounded to a radius of about 0.003 in. so they would fit into the punched gage marks in the manner shown in Fig. 2. It will be noted that the point of the dividers rests on the sloping sides of the gage mark and does not touch the bottom. This results in greater accuracy in locating the dividers in the punch mark. After the dividers had been set for the final distance between gage marks they were placed on a steel scale with one point of the dividers at an even inch mark. The scale was divided into fiftieths of an inch, thus permitting it to be read to one hundredth inch very easily without the eye strain associated with reading a scale divided into hundredths of an inch.

First Series:

In the first series of tests there were 305 test bars of alloy CN21. Two hundred and three of these were cast with grip ends 2 in. long and 102 with ends 3 in. long. The ends of half the bars with 3-in. ends were cut to a length of 1 in.

The reduced section of each test bar was filed where necessary to remove the parting line irregularities. Centers were drilled in the ends of each bar while the reduced section was held in a precision scroll-type lathe chuck to make the centers concentric with the reduced section. The ends of half the bars of each group were machined so as to make them cylindrical. The ends of the remaining bars were merely cleaned up slightly by filing off the parting lines and other irregularities.

Second Series:

The second series consisted of 106 bars from a second melt of alloy CN21. The preparation of the bars with 1-in. long ends was similar to that of the first series. The reduced sections of all the bars with 2-in. ends were machined just enough to true them up thoroughly. The ends of half these bars were machined cylindrical and concentric with the reduced sections; the ends of the remaining bars were threaded to fit the ¾-in. 10 threads of the adapters. The threads were chased with a lathe tool while the specimen was on centers, thus making the threads concentric with the axis of the specimen.

TABLE I.—RESULTS OF TENSION TESTS OF SAND-CAST ALUMINUM ALLOY CN21 TEST BARS.

Test Bar Ends		Number of Tests	Tensile Strength, psi.			Elongation in 2 in., per cent		
Length, in.	Preparation		Maximum	Minimum	Average	Maximum	Minimum	Average
			FIRST SERIES					
1....	Not machined	26	36 000	29 000	32 700	1.0	0.5	0.7
	Machined	25	35 700	27 700	33 000	1.0	0.5	0.7
2....	Not machined	101	37 200	29 300	32 950	1.0	0.0	0.6
	Machined	99	36 700	28 200	33 200	1.0	0.0	0.6
3....	Not machined	26	36 600	29 100	33 300	1.0	0.5	0.6
	Machined	25	35 100	29 500	32 000	1.0	0.5	0.8
			SECOND SERIES					
1....	Not machined	25	38 500	31 400	35 500	1.0	0.0	0.4
	Machined	25	37 300	29 300	35 000	1.0	0.0	0.3
2....	Machined[a]	28	38 900	30 200	35 000	1.0	0.0	0.4
	Threaded ends[a]	28	38 800	29 700	35 100	1.0	0.0	0.4

[a] Reduced sections of test bars machined to remove cast surface.

RESULTS OF TESTS

The data from these two series of tests of alloy CN21 bars are summarized in Table I. It seems evident from the results of the first series that neither the length of the grip ends nor the machining of the cylindrical surfaces of the ends had any significant effects upon the test results. The second series of tests confirms this and shows, furthermore, that machining the reduced sections and using threaded ends made no difference in the elongation values or the other tensile properties.

There are, however, some facts that cannot be overlooked. In the first place, all bars had been radiographed for soundness and carefully inspected for straightness. If the bars had not been as straight as they were the variations in results would almost certainly have been greater. Furthermore, the bars were carefully prepared and were tested with great care. In each test, for instance, the shoulders at the ends of the test bars were placed even with the ends of the V-grips, except that when the ends were 3 in. long the shoulders were about ½ in. outside the grips. This insured about as good gripping as possible. Of perhaps greater importance as far as the measurement of elongation is concerned is the fact that the device used for holding the broken bars together assured practically perfect fitting of the two parts. If all these precautions had not been taken, it is quite likely that the results would have shown much greater variation. It is also believed that under less favorable conditions of gripping, the test bars with short ends would have given less satisfactory results.

TABLE II.—RESULTS OF TENSION TESTS OF SAND-CAST ALUMINUM ALLOY C1 TEST BARS HAVING DIFFERENT DIMENSIONAL CHARACTERISTICS.

Test Bar Ends		Number of Tests	Tensile Strength, psi.			Yield Strength[a], psi. (Average)	Elongation in 2 in., per cent		
Length, in.	Preparation		Maximum	Minimum	Average		Maximum	Minimum	Average
1	Not machined	50	40 300	32 100	36 500	26 900	5.0	2.0	3.0
	Machined	50	41 700	33 800	36 700	27 200	5.0	1.5	3.0
2	Not machined	25	39 000	34 200	37 400	27 100	4.0	2.0	3.3
	Machined	25	39 000	33 500	36 800	27 300	4.0	2.0	2.9
3	Not machined	50	39 900	33 600	36 600	27 300	4.0	2.0	3.0
	Machined	50	40 000	33 000	36 600	27 100	4.5	2.0	3.1

[a] Offset equals 0.2 per cent.

The results of the tests of alloy C1 bars, which are summarized in Table II, show much the same thing that the tests of alloy CN21 showed. Alloy C1 has considerably more elongation and for that reason might be expected to be less sensitive to the factors under consideration.

The measurement of elongation, with which we were primarily concerned in planning the investigation, was given more attention than the determination of tensile strength and yield strength. Alloy CN21 has a nominal elongation in 2 in. of about 0.5 per cent. In these tests the measured values ranged from zero to 1.0 per cent. The average values of elongation for each group of specimens determined independently by two individuals showed maximum differences of only 0.1 per cent, but considering the bars separately, there were many instances where the values determined by these two experienced individuals differed by 0.5 per cent. Both of these men were able to match the broken halves together, place them in the holder and confirm their previous measurements within 0.5 per cent, but even with repeated measurements the two individuals differed in many cases by 0.5 per cent. These specimens all broke with clean square fractures so that it was an easy matter to match the two halves of the specimens together. If the material had been such as to cause irregular, jagged fractures it would have been much more difficult to match the two parts together, and for that reason the elongation values might have shown greater variation.

In the tests of alloy C1, which has a nominal elongation in 2 in. of 3 per cent, values for individual bars ranged from 1.5 to 5.0 per cent, and the average of each group was quite close to 3.0 per cent. Measurements of elongation could be repeated within 0.5 per cent in all cases, but here again measurements of the same bar by the two individuals often differed by 0.5 per cent.

TABLE III.—COMPARISON OF ELONGATION VALUES OBTAINED USING DIFFERENT MEASURING DEVICES.

Test Bar	Micrometer Filar Microscope	Dividers and Scale		Vernier Calipers		Dial Gage Instrument	
	Elongation, per cent	Elongation, per cent	Difference[a]	Elongation, per cent	Difference[a]	Elongation, per cent	Difference[a]
No. 1......	1.27	1.5	−0.23	1.25	−0.02	1.4	0.13
No. 2......	0.96	1.0	0.04	0.95	−0.01	1.0	0.04
No. 3......	1.56	1.5	−0.06	1.55	−0.01	1.5	−0.06
No. 4......	2.26	2.0	−0.26	2.25	−0.01	2.3	0.04
No. 5......	2.58	2.5	−0.08	2.55	−0.03	2.6	0.02
No. 6......	1.50	1.5	0.00	1.60	0.10	1.6	0.10
No. 7......	1.44	1.5	0.06	1.45	0.01	1.5	0.06
No. 8......	0.64	0.5	−0.14	0.65	0.01	0.7	0.06
No. 9......	0.60	0.5	−0.10	0.65	0.05	0.6	0.00
No. 10......	0.01	0.0	−0.01	0.00	−0.01	0.0	−0.01
Average...	1.282	1.25		1.290		1.32	

[a] Difference from value determined using micrometer filar microscope.

These results make it quite clear that even under conditions much better than usually exist in ordinary routine tests, there is no justification for attempting to measure elongation values closer than 0.5 per cent in 2 in. when using dividers and scale.

Realizing that there might be more precise ways of measuring the final distance between gage marks, ten bars were chosen at random to be measured by three other methods as well as by dividers and scale (Table III). The first of these, and what might be considered the most precise, was by means of a micrometer filar microscope reading directly to 0.01 mm. (0.004 in.) and by estimation to 0.002 mm. (0.0008 in.). The values of elongation determined by the use of dividers and scale in this series of measurements agreed in all cases with those measured with the filar microscope within 0.26 per cent in 2 in.; the average difference between the elongation values determined by the two methods was 0.03 per cent in 2 in. Measurements made with vernier calipers reading to 0.001 in. agreed in all cases with those determined by dividers and scale within 0.25 per cent in 2 in., and the averages differed by 0.1 per cent in 2 in. The agreement is very satisfactory, since no attempt was made to estimate the readings made with dividers and scale to closer than 0.5 per cent.

The third set of measurements was made using an elongation measuring instrument provided with a dial indicator in which one division equalled 1 per cent elongation in 2 in. Readings taken with this gage, estimating tenths of a division, could not be repeated consistently closer than 0.5 per cent in 2 in., which is about the same as with dividers and scale. The average of several measurements of each of the ten bars is shown in Table III. It was our experience that more time was required to measure elongation with this instrument than with dividers and scale.

SUMMARY

These tests show that with straight test bars, carefully prepared and carefully tested following a good testing technique, elongation values determined by two experienced individuals on the same test bar are very likely to differ by 0.5 per cent in 2 in. Without such careful attention to details, values would not agree this closely. In ordinary commercial laboratory inspection testing, it is impractical to give as much consideration to the details of testing as was done in these tests. The time required to make the tests and therefore the cost of testing would be excessive. Furthermore, the caliber of the individuals making routine inspection tests is generally not as high as that of the individuals conducting these investigational tests.

Specifications now require that elongation in 2 in. be measured to 0.5 per cent in 2 in., and these tests indicate quite definitely that it would be unreasonable to expect such values to be determined any more closely.

REPORT OF COMMITTEE B-8*
ON
ELECTRODEPOSITED METALLIC COATINGS

Committee B-8 on Electrodeposited Metallic Coatings held two meetings during the year: in Pittsburgh, Pa., on March 1, 1946, and in Philadelphia, Pa., on February 28, 1947.

The resignation of R. J. McKay as chairman was accepted with regret at the February meeting of the Advisory Committee and C. H. Sample of the International Nickel Co., Inc., was appointed chairman for the unexpired term of Mr. McKay, and R. B. Saltonstall of the Udylite Corp., was appointed secretary for the unexpired term of Mr. Sample.

Recommendations Affecting Standards

The committee recommends that the standard and all tentatives under its jurisdiction be continued in their present status without revision pending results of further research and development work in the field of electrodeposited metallic coatings currently being carried on by the committee, the Research Committee of the American Electroplater's Society, and others.

Activities of Subcommittees

Subcommittee I on Specifications, Papers, and Definitions (K. G. Soderberg, chairman) secured for presentation at the 1947 Annual Meeting the following three technical papers on inspection and rating of various electroplated exposure test panels: "Rating Exposure Test Panels of Decorative Electrodeposited Cathodic Coatings,"[1] by W. A. Wesley; "Inspection of Exposure Test Panels with Nondecorative Electrodeposited Cathodic Coatings,"[2] by H. A. Pray; and "Inspection of Exposure Test Panels with Nondecorative Electrodeposited Anodic Coatings,"[3] by K. G. Soderberg. At the March meeting of the subcommittee, J. E. Stareck of United Chromium, Inc., was elected chairman to take office at the close of the Annual Meeting.

The subcommittee has voted to clarify the definition of nickel (*versus* nickel-cobalt alloys) as used in the several specifications covering electrodeposited metallic coatings under its jurisdiction by editorially inserting a footnote to read as follows, "The term nickel as used in these specifications includes nickel-cobalt alloys in which the nickel is the major constituent." This footnote will be added to the table in Section 3 of the following three Specifications for Electrodeposited Coatings of Nickel and Chromium: on Steel (A 166)[4], on Copper and Copper-Base Alloys (B 141)[4], and on Zinc and Zinc-Base Alloys (B 142)[4].

Subcommittee II on Performance Tests (H. A. Pray, chairman) has conducted exploratory exposure tests of copper-nickel-chromium coatings on steel and has continued its tests of electrodeposited lead coatings on steel as covered in the report of this subcommittee which is appended hereto.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] See p. 803.

[2] See p. 822.

[3] See p. 826.

[4] 1946 Book of A.S.T.M. Standards, Part I-B.

Subcommittee III on Conformance Tests (W. A. Wesley, chairman) has organized the following working sections: I on Thickness Tests (A. H. Du Rose, chairman), II on Porosity Tests (A. Mendizza, chairman), III on Adhesion, Hardness and Ductility Tests (E. J. Roehl, chairman). A fourth section on luster tests is in the course of organization. These sections are maintaining close liaison with corresponding active research projects of the American Electroplater's Society. Pending the results of the work on thickness tests the subcommittee recommends that the Tentative Methods of Test for Local Thickness of Electrodeposited Coatings (A 219 - 45 T) be retained as tentative.

Subcommittee IV on Electroplating Practice (A. K. Graham, chairman).—A subgroup (R. M. Wick, chairman) has prepared a proposed Recommended Practice for the Preparation of High-Carbon Steel for Electroplating which will be submitted to the committee for letter ballot action in the near future. At the March meeting of the subcommittee the following officers were elected for the term beginning with the close of the Annual Meeting: chairman, F. K. Savage; vice-chairman, M. B. Diggin; secretary, K. M. Huston.

Subcommittee V on Supplementary Protective Finishes for Metallic Coatings (Marc Darrin, chairman).—This subcommittee recommends continuation of the Tentative Specifications for Chromate Finishes on Electrodeposited Zinc, Hot-Dipped Galvanized, and Zinc Die-Cast Surfaces (B 201 - 45 T) pending the results of tests which are currently being formulated.

This report has been submitted to letter ballot of the committee which consists of 80 voting members; 63 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

C. H. SAMPLE,
Chairman.

R. B. SALTONSTALL,
Secretary.

REPORT OF SUBCOMMITTEE II ON PERFORMANCE TESTS

ATMOSPHERIC EXPOSURE OF COPPER-NICKEL-CHROMIUM DEPOSITS ON HIGH-CARBON STEEL

The exposure tests at New York, N. Y., Pittsburgh, Pa., and Kure Beach, (Wilmington), N. C., involving an exploratory series of panels representing various combinations of copper, nickel, and chromium on high-carbon steel, have been continued. The tests are serving their intended purpose, which was primarily to indicate the efficacy of the plating techniques and to develop an inspection method. Based on the results of the exploratory tests, a more extensive series of coatings of the same type has been prepared and recently exposed at five locations. Details of the methods employed and the results obtained will be described in a subsequent report.

The exploratory tests tentatively indicate that the atmospheric life of the composite coatings on high-carbon steel is not greatly influenced by the thickness of the copper layer and that the determining factor is the thickness of the nickel. Confirmation of these conclusions awaits the results of the more extensive tests now in progress.

ATMOSPHERIC EXPOSURE OF ELECTROPLATED LEAD COATINGS ON STEEL

The more recent results of inspections of the specimens of electroplated lead coatings on steel, exposed to the atmosphere at New York, N. Y., State College, Pa., Kure Beach (Wilmington), N. C., and Tela, Honduras, are summarized in Table I. This table is a continuation of Table IV of the 1944 report,[1] Table I of the 1945 and 1946 reports.[1]

The method of recording the data described in the subcommittee report for 1946 has been continued. It is becoming increasingly difficult, especially at New York, to make an absolute distinction between the "S" (percentage of area stained, coating intact) and the "R" (percentage of flaky rust of base metal) ratings. An uncertainty, therefore, exists in the visual estimation of the area of a panel on which the coating has retained some protective value or the area on which active corrosion of the steel base has started. The ratings given in Table I involve a few discrepancies resulting from differences in the inspectors' judgment from time to time on this point and must be regarded as semiquantitative at best. They are intended to give a general idea of the average appearance of the panels and an indication of the relative protectiveness of the various coatings in so far as it can be estimated visually.

The trends indicated at the earlier stages of the exposure appear to be continuing. Electroplated lead coatings are generally more protective to steel in the industrial atmosphere of New York and less so in the seacoast atmosphere of Kure Beach than at the other stations. The reverse appears to be true of the zinc-coated control panels. As previously indicated, apparent damage to the steel base increases progressively with exposure time and decreases with increasing coating thickness. The some-

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[1] *Proceedings*, Am. Soc. Testing Materials, Vol. 44, p. 280 (1944), Vol. 45, p. 152 (1945), and Vol. 46, p. 266 (1946), respectively.

TABLE I.—SUMMARY OF INSPECTION DATA ON THE ATMOSPHERIC EXPOSURE TESTS OF ELECTROPLATED LEAD COATINGS.

	Nominal Coating Thickness, in.	Number of Panels Averaged, Each Station	New York, N. Y.				State College, Pa.			
			Upper Surface		Under Surface		Upper Surface		Under Surface	
			2.40 yr.	2.74 yr.	2.40 yr.	2.74 yr.	2.28 yr.	2.63 yr.	2.28 yr.	2.63 yr.
Sulfamate lead on steel	0.00050	5	MP-30S	MP-40S	MP-45S	MP-10S	MP-85S-10R	MP-100S-55R	2P	25P
	0.00100	5	....	MP	MP-1S	MP	5P	MP<1S	....	....
Fluoborate lead on steel	0.00008	3	MP-100S-40R	100R	MP-100S-8R	MP-95S-5R	100R	100R	100R	100R
	0.00025	5	MP-100S	MP-100S	MP-95S-1R	MP-65S	100R	100R	100R	MP-100S-60R
	0.00050	5	MP-90S	MP-90S	MP-60S	MP-30S	MP-90S-40R	MP-100S-50R	20P-4S	MP-2S
	0.00075	5	MP-75S	MP-70S	MP-55S	MP-30S	MP-5S	MP-60S-7R	....	....
	0.00100	5	MP-15S	MP-25S	MP-25S	MP-8S	MP	MP-2S	....	....
	0.00200	17	....	....	2P	....	....	....	....	....
Sulfamate lead on copper-plated steel	0.00050	5	2P	MP	MP-15S	MP-3S	MP-5S	MP-10S-1R	10P	MP-1S
	0.00100	5	....	....	2P	....	1P	MP	5P	4I
Fluoborate lead on copper-plated steel	0.00008	3	100R	100R	MP-95S-5R	MP-90S-7R	100R	100R	100R	100R
	0.00025	5	MP-5S	MP-15S	MP-20S	MP-5S	MP-40S-10R	MP-100S-70R	MP-20S	MP-15S-1R
	0.00050	5	....	MP	MP-5S	MP-1S	15P	MP<1S	15P	MP<1S
	0.00075	5	....	MP	MP-1S	MP-1S	MP	MP<1S	1P	3P
	0.00100	5	....	....	2P	MP	20P	MP<1S	3P	2P
	0.00200	17	....	....	....	....	....	....	....	....
Sulfamate lead on sheet copper	0.00025	3	....	....	....	....	....	....	....	....
	0.00050	3	....	....	....	....	....	....	....	....
Fluoborate lead on sheet copper	0.00025	3	....	....	....	....	....	....	....	....
	0.00050	3	....	....	....	....	....	....	....	....
Sulfamate lead-tin on steel										
2 per cent tin	0.00100	3	1P	MP-30S	MP-10S	MP-5S	MP-100S-75R	MP-100S-70R	MP-40S	MP-40S-3R
10 per cent tin	0.00100	3	....	MP-3S	2P	....	MP-90S-30R	MP-100S-50R	....	....
Commercial hot-dipped terne (20 per cent tin)	0.00017	3	MP-100S	MP-100S	MP-50S	MP-25S	100R	100R	100R	100R
Commercial hot-dipped lead (2.5 per cent tin, 2 per cent antimony)	0.00040	3	MP-90S-5R	MP-75S	MP-40S	MP-20S	MP-98S-60R	MP-100S-60R	100R	MP-100S-70R
Electroplated zinc on steel	0.00020	3	100R	100R	100R	100R	MP-50S-30R	70R	....	....
Hot-dipped zinc on steel	0.00075	3	MP-90S-45R	100R	MP-20S	30S-15R	20P-10S	....	....	....
Bare steel	....	6	100R	100R	100R	100R	100R	100R	100R	100R
Fluoborate lead on steel (damaged)	0.00050	3	Sl. spread	Sl. spread	MP-10S	MP-2S	Bad spread	Bad spread	MP-10S	MP-85-1R
	0.00100	3	Sl. spread	Sl. spread	....	....	Sl. spread	Sl. spread	1P	1P
Fluoborate lead on copper-plated steel (damaged)	0.00050	3	Sl. spread	Sl. spread	....	MP	Sl. spread	Spread	10P	20P
	0.00100		Sl. spread	Sl. spread	....	MP	V. sl. spread	Sl. spread	5P	....

TABLE I.—*Continued*

	Nominal Coating Thickness, in.	Number of Panels Averaged Each Station	Kure Beach, N. C.				Tela, Honduras			
			Upper Surface		Under Surface		Upper Surface		Under Surface	
			1.34 yr.	2.38 yr.	1.34 yr.	2.38 yr.	1.83 yr.	2.27 yr.	1.83 yr.	2.27 yr.
Sulfamate lead on steel	0.00050	5	MP-100S	MP-100S	MP-50S	MP-95S-2R	15R	MP-40S-20R	15R	MP-50S-25R
	0.00100	5	MP-3S	MP-20S	MP<1S	MP-2S	15P	MP	10P	MP
Fluoborate lead on steel	0.00008	3	100R	100R	100R	100R	100R	100R	100R	100R
	0.00025	5	MP-100S-60R	100R	MP-100S-60R	98R	95R	100R	85R	100R
	0.00050	5	MP-100S-1R	MP-100S	MP-90S-3R	MP-100S	30R	MP-45S-25R	30R	MP-40S-40R
	0.00075	5	MP-100S-1R	MP-100S	MP-65S	MP-90S	1P	MP-5S	5R	MP-20S-10R
	0.00100	5	MP-60S	MP-85S	MP-25S	MP-50S	MP	MP	MP	MP-5S
	0.00200	17	25P	MP	10P	MP	8P	MP	1P	2P
Sulfamate lead on copper-plated steel	0.00050	5	MP-80S	MP-98S	MP-25S	MP-50S	1P	MP	5R	MP-10S-5R
	0.00100	5	MP-1S	MP-5S	MP<1S	MP-2S	10P	10P	2P	MP
Fluoborate lead on copper-plated steel	0.00008	3	100R	100R	100R	100R	100R	100R	100R	100R
	0.00025	5	MP-80S-20R	MP-100S-70R	MP-100S 75R	MP-100S-65R	45R	MP-70S-60R	60R	MP-80S-70R
	0.00050	5	MP-25S	MP-70S	MP-25S	MP-25S	MP	MP-15S-3R	10R	MP-20S-12R
	0.00075	5	MP-30S	MP-50S	MP-1S	MP-3S	20P	MP	MP	MP
	0.00100	5	MP-12S	MP-20S	MP-5S	MP-8S	15P	20P	20P	MP
	0.00200	17	15P	MP	10P	MP	10P	10P	1P	2P
Sulfamate lead on sheet copper	0.00025	3	....	....	....	....	....	....	....	....
	0.00050	3	....	....	....	....	....	....	....	....
Fluoborate lead on sheet copper	0.00025	3	....	....	....	....	....	....	....	....
	0.00050	3	....	....	....	....	....	....	....	....
Sulfamate lead-tin on steel:										
2 per cent tin	0.00100	3	MP-100S	MP-100S	MP-90S	MP-95S	85R	MP-100S-80R	55R	MP-85S-65R
10 per cent tin	0.00100	3	MP-100S	MP-100S-15R	MP-30S	MP-20S	50R	MP-70S-40R	40R	MP-50S-40R
Commercial hot-dipped terne (20 per cent tin)	0.00017	3	MP-100S-80R	MP-100S-95R	MP-100S-60R	MP-100S-75R	90R	95R	80R	80R
Commercial hot-dipped lead (2.5 per cent tin, 2 per cent antimony)	0.00040	3	MP-100S-45R	MP-100S-40R	MP-100S-30R	MP-100S-30R	60R	MP-75S-65R	55R	MP-75S-70R
Electroplated zinc on steel	0.00020	3	50R	100R	....	MP	40R	75R	MP	20R
Hot-dipped zinc on steel	0.00075	3	—..	....	....	....	MP	6P	....	....
Bare steel	....	6	100R	100R	100R	100R	100R	100R	100R	100R
Fluoborate lead on steel (damaged)	0.00050	3	Def. spread	V. bad spread	MP-80S-2R	MP-100S	....	....	60R	MP-85S-75R
	0.00100	3	Spread	Bad spread	25P	MP	....	....	1P	MP
Fluoborate lead on copper-plated steel (damaged)	0.00050	3	Def. spread	Bad spread	MP-25S	MP-60S	....	....	5R	15R
	0.00100		Sl. spread	Sl. spread	5P	MP	....	....	10S	MP-5S

Abbreviations and symbols used: P—number of pinholes, up to 25; MP—pinholes, more than 25; IP—pinholes, more than 25, irregularly distributed; RP—pinholes, more than 25, distributed uniformly; R—percentage of flaky rust of base metal; S—percentage of area stained, coating intact; Sl.—slight; Def.—definite.
NOTE.—Thickness of copper plate in all cases, 0.000015 in.

what superior performance of lead de-deposits over a copper flash (0.000015 in.) on steel persists as the appearance of deterioration progresses to the thicker coatings. As noted previously, the rural and seacoast atmospheres produce a white corrosion product (most evident on the under surface) on the panels plated with pure lead and not on the lead alloy-coated specimens. At State College and Kure Beach, the lead-on-copper sheet panels are acquiring a purplish tint which may be due to the presence of copper corrosion product on the coating surface.

The subcommittee plans to remove selected panels during the coming year and to determine the damage to the base metal by measuring losses in weight and tensile strength. The data obtained should be useful in verifying the tentative conclusions based on visual examination.

For the purpose of determining the rate at which electrodeposited lead itself corrodes in the diverse atmospheres represented in the tests, panels provided with the heaviest coating (0.002 in.) have been removed from the racks, stripped of corrosion products and examined by the weight-loss method.

The lead corrosion products were removed by the following method. A solution of equal volumes of water and ammonia (28.5 per cent by weight) was first neutralized with 80 per cent acetic acid and to this 2 ml. of 80 per cent acetic acid per 100 ml. of solution was added. The panels were immersed in this solution for 5 min. at 130 to 140F. They were

TABLE II.—ATMOSPHERIC CORROSION OF ELECTROPLATED LEAD.

Designation Symbol[a]	Specimen Number	Initial Total Weight, g.	Weight of Deposit, g.	Weight as Received Before Cleaning, g.	Weight After Cleaning, g.	Loss in Weight, g.	Initial Average Thickness, in.	Average Loss in Thickness, in.	Average Penetration, in. per yr.
STATE COLLEGE, PA., 2.28-YR. EXPOSURE									
C-20-F-66	474	129.79	17.56	129.53	128.26	1.53	0.00196	0.000171	....
C-20-F-67	475	125.80	17.07	125.53	124.20	1.60	0.00191	0.000179	....
C-20-F-62	470	127.76	16.68	127.52	126.21	1.55	0.00187	0.000174	....
S-20-F-62	230	124.35	16.94	124.06	122.99	1.36	0.00190	0.000152	....
S-20-F-66	234	125.30	17.49	125.05	123.86	1.44	0.00196	0.000161	....
S-20-F-67	235	129.88	17.11	129.62	128.29	1.59	0.00192	0.000178	0.000074
KURE BEACH, N. C., 2.38-YR. EXPOSURE									
S-20-F-63	231	129.67	17.46	129.34	127.95	1.72	0.00196	0.000193	....
S-20-F-65	233	125.06	17.22	124.73	123.24	1.82	0.00193	0.000204	....
S-20-F-70	238	124.38	16.28	124.04	122.68	1.70	0.00183	0.000191	....
C-20-F-29	437	124.30	16.66	124.00	122.33	1.97	0.00186	0.000220	....
C-20-F-63	471	128.82	17.25	128.49	126.77	2.05	0.00193	0.000229	....
C-20-F-65	473	128.67	16.89	128.29	126.69	1.98	0.00189	0.000222	0.000088
NEW YORK, N. Y., 2.4-YR. EXPOSURE									
S-20-F-61	229	124.54	17.01	124.53	123.95	0.59	0.00191	0.000066	....
S-20-F-68	236	127.44	17.14	127.41	126.86	0.58	0.00192	0.000065	....
S-20-F-72	240	127.53	16.71	127.50	126.95	0.58	0.00187	0.000065	....
C-20-F-61	469	126.24	16.78	126.23	125.64	0.60	0.00188	0.000067	....
C-20-F-68	476	128.52	17.15	128.49	127.93	0.59	0.00192	0.000066	....
C-20-F-72	480	127.00	16.81	126.99	126.43	0.57	0.00188	0.000064	0.000033
TELA, HONDURAS, 2.27-YR. EXPOSURE									
S-20-F-64	232	124.43	17.02	124.42	122.95	1.48	0.00191	0.000166	....
S-20-F-69	237	128.35	17.68	128.35	126.92	1.43	0.00198	0.000160	....
C-20-F-64	472	130.01	16.91	130.04	128.48	1.53	0.00190	0.000172	....
C-20-F-69	477	128.39	17.86	128.46	126.70	1.69	0.00200	0.000189	....
C-20-F-71	479	127.09	16.41	127.08	125.64	1.45	0.00184	0.000163	0.000075

[a] Indicates panels plated with approximately 0.002 in. of fluoborate lead either on bare steel (S) or copper plated steel (C).

then swabbed with absorbent cotton, rinsed, dried, and weighed. This procedure, applied to unexposed panels of the same type, resulted in negligible weight loss. No appreciable damage occurred to the steel base of the exposed panels, as evidenced by a very minor amount of pinholing at the time of their removal. The weight loss of the stripped panels, therefore, provides a basis for estimation of the corrosion rate of the electroplated lead coating. It should be noted, however, that the rate so obtained is an average rate for the upper and under surfaces which obviously were subjected to different exposure conditions. The results are given in Table II.

The data show that the rate of weathering of electrodeposited lead is very closely the same at State College and Tela (0.000074 and 0.000075 in. per year respectively), significantly greater at Kure Beach (0.000088 in. per year), and much lower in the relatively high sulfur-containing atmosphere of New York (0.000033 in. per year). The rate of corrosion was slightly higher when the lead was deposited over the copper plate than when directly on the steel. These rates bear an inverse relation to the protective value of the coatings exposed to the same atmospheres. Further, the depth of penetration per year is of the same order as the coating thickness for the lightest coatings tested. It is, therefore, not surprising that the thin coatings have proven to be protective for relatively short periods of time.

Respectfully submitted on behalf of the subcommittee,

H. A. PRAY,
Chairman.

REPORT OF COMMITTEE B-9[*]
ON
METAL POWDERS AND METAL POWDER PRODUCTS

This report covers the activities of Committee B-9 on Metal Powders and Metal Powder Products for the period from June, 1946, to June, 1947. During this time, two meetings were held: in Buffalo, N. Y., on June 28, 1946, and in New York, N. Y., on March 21, 1947.

RECOMMENDATIONS AFFECTING STANDARDS

The committee recommends that the proposed Specifications for Sintered Metal Powder Structural Parts[1] be accepted for publication as tentative. This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

During the March 21, 1947, meeting of Committee B-9 in New York, N. Y., it was unanimously agreed by the committee that the Tentative Specifications for Metal Powder Sintered Bearings (Oil Impregnated) (B 202 - 45 T) should be continued as tentative because of projected additions to the appendix to these specifications as a result of work now in progress.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Technical Data (F. N. Rhines, chairman).—The glossary which now includes 131 terms used in powder metallurgy has been fully agreed on by the subcommittee and is now out for committee ballot.

This subcommittee has submitted to the Advisory Committee, for consideration, possible tasks for the future as follows:

1. To administer the glossary by editing all publications of Committee B-9 with respect to terminology,
2. To finish the work on standard tension test bars for use with metal powders and develop such other specimens that might be required,
3. To prepare tabulations of technical data when and if such are required, and
4. To prepare and maintain a well-indexed bibliography on powder metallurgy if this is required.

These matters will be taken up at the next Advisory Committee meeting and action recommended.

Subcommittee II on Metal Powders (D. O. Noel, chairman) is actively engaged in working out test methods for chemical analysis of metal powders, and close cooperation from Committee E-3 on Chemical Analysis of Metals is proving very helpful. The subcommittee is also working on the standardization of several methods for determining subsieve particle size and on a test for compressibility of metal powders. Much of this work is being carried on as a cooperative program in the laboratories of the subcommittee members.

Subcommittee III on Metal Powder Products (R. P. Koehring, chairman): Section 1 on Bearings (D. S. Urquhart, chairman) has been pursuing the development of a standard size list for bearings including press fit and recommended clearances.

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] These specifications were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-B.

[2] The letter ballot on this recommendation was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Section 2 on Structural Parts (F. V. Lenel, chairman) has developed the proposed Specifications for Sintered Metal Powder Structural Parts, referred to earlier in this report. A report by the special committee on high-density iron and iron-graphite structural parts indicated there was insufficient interest in a specification for these materials at present.

Section 3 on Cemented Carbides (M. F. Judkins, chairman) is working on test methods for transverse rupture, hardness, chemical composition, and microstructure. It is also considering the desirability of a classification and designation system for the various grades of commercially produced carbides.

Section 4 on Electrical Parts (A. B. Gibson, chairman)—The work of this section is centered about two main products, pole pieces and electrical contacts. The proposed Ring Test Method for Magnetic Properties of Pole Pieces, which has been approved by Committee B-9, is to be submitted to Committee A-6 on Magnetic Properties. Regarding electrical contacts, methods for determining resistance, hardness, density, and tensile strength are under consideration.

Section 5 on Friction Materials (J. R. Nurney, chairman).—As noted in the 1946 Report[3] of Committee B-9, this section continues simply as a medium for exchange of information on friction materials with a view to engaging in standardizing work as soon as it becomes advisable.

This report has been submitted to letter ballot of the committee, which consists of 50 members; 39 members returned their ballots, of whom 35 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. A. REICH,
Chairman.

W. R. TOEPLITZ,
Secretary.

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 270 (1946).

REPORT OF JOINT COMMITTEE*

ON

FILLER METAL

The Joint A.W.S.-A.S.T.M. Committee on Filler Metal, through its subcommittees, has continued the formulation of standards for ferrous and nonferrous filler metal. During the past year, the joint committee presented to A.S.T.M. through its Administrative Committee on Standards the recommendation that the Tentative Specifications for Iron and Steel Gas-Welding Rods (A.S.T.M. A 251 - 42 T; A.W.S. A 5.2 - 46 T) be revised. This recommendation was approved by the A.W.S. Board of Directors on June 20, 1946 and accepted by the A.S.T.M. Standards Committee on July 26, 1946.

On October 26, 1946, the A.W.S. Board of Directors approved the new Tentative Specifications for Corrosion-Resisting Chromium and Chromium-Nickel Steel Welding Electrodes (A.S.T.M. A 298 - 46 T; A.W.S. A 5.4 - 46 T). These specifications were accepted by the A.S.T.M. Standards Committee on November 23, 1946.

The new and the revised specifications appear in the 1946 Book of A.S.T.M. Standards, Part I-A.

Several additional specifications are now under consideration and will be submitted for approval shortly. These are indicated in the reports of the respective subcommittees which follow. It will be noted that Subcommittee VIII on Capillary Brazing Materials and Subcommittee X on Classification Designation of Filler Metal have been organized during the past year.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Iron and Steel Arc-Welding Electrodes (J. H. Deppeler, chairman) held meetings on September 19 and 20, 1946, at which time it was suggested that the Tentative Specifications for Iron and Steel Arc-Welding Electrodes (A.S.T.M. A 233 - 45 T; A.W.S. A 5.1 - 45 T) consist of three parts and a subcommittee was appointed to make this division. It is believed that most of the necessary revisions have been discussed and that, in the near future, the revised specifications will be submitted to the main committee for letter ballot vote.

Subcommittee II on Iron and Steel Gas Welding Rods (J. H. Critchett, chairman) prepared the revision of Tentative Specifications A 251 mentioned earlier in this report. Copies of the revised specifications are now being circulated among users in industry to determine whether any further changes are desirable. Future activity of the subcommittee will be based on the recommendations received.

Subcommittee III on Aluminum and Aluminum-Alloy Filler Metal (G. O. Hoglund, chairman) has been inactive pending completion of tests to determine the mechanical properties of aluminum and aluminum-alloy gas-welding rods. It is expected that the subcommittee will review the data obtained and will proceed to complete the preparation of specifications for aluminum and aluminum-alloy gas-welding rods. When this has been completed, consideration will be given to revision of the Tentative Specifications for Aluminum and Aluminum-Alloy Metal Arc-Welding Electrodes (A.S.T.M. B 184 - 43 T; A.W.S. A 5.3 - 43 T).

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

Subcommittee IV on High-Alloy Steel Filler Metal (R. D. Thomas, chairman) prepared the Tentative Specifications A 298 referred to earlier. Appended to these specifications is a guide explaining the method of classifications in accordance with the specifications and the applications of the various grades included therein. It is believed that the issuance of these specifications will be a valuable contribution in standardizing corrosion-resistant electrodes.

Since this is the first issue of specifications for this category of materials, it is anticipated that the need for revisions will become apparent within a year after publication. Already certain grades of alloy electrodes might well be added to make the specifications more complete.

Subcommittee V on Nickel and Nickel-Alloy Filler Metal (O. B. J. Fraser, chairman) has been reorganized during the past year. Draft specifications are now being prepared.

Subcommittee VI on Copper and Copper-Alloy Filler Metal (C. E. Swift, chairman) is continuing the preparation of specifications for copper and copper-alloy electrodes. A special group has been appointed to review the electrode requirements in detail and report back to the subcommittee. As soon as these specifications are completed, work will be started on similar specifications for gas welding rods.

Subcommittee VII on Surfacing Materials (R. K. Lee, chairman) has functioned under the chairmanship of M. G. Sedam until February 4, 1947, at which time Mr. Sedam resigned and R. K. Lee was elected chairman.

According to action previously reported, the members of the subcommittee furnished chemical analyses of weld metal from all types and from all manufacturers of electrodes. A working group met and arranged those analyses according to the outline of composition prepared at the March 26, 1946, meeting. This arrangement of the analyses was distributed to the members of the subcommittee on November 18, 1946.

A meeting of the subcommittee, held in York, Pa., on February 4, 1947, was attended by seven members. At that meeting generalities concerning the specifications were discussed. It was agreed to continue the classification of the surfacing materials according to chemical analysis, without reference in the specifications proper to abrasion resistance, impact properties, or other specific properties. Such information will be attached to the specifications as a guide. The arrangement of the analyses which had previously been distributed was discussed in detail with changes, such as new divisions, being made where necessary.

Another meeting of the subcommittee was held March 24, 1947, in New York, N. Y., at which time the detailed discussion of the classification of the analyses were completed. It is proposed to have frequent meetings of the subcommittee, at least every two months, until the specifications are completed.

Subcommittee VIII on Capillary Brazing Materials (J. L. Christie, chairman) has just been organized. Plans are being made for the first meeting.

Subcommittee X on Classification Designation of Filler Metal (R. David Thomas, Jr., chairman) was appointed in May, 1946, to prepare a standard method of designating all filler metal. The subcommittee has held three meetings and has discussed all aspects of the problem. The discussion has resolved the problem into two viewpoints; one in which the designating number gives certain important information regarding the characteristics of the filler metal, and the other in which the designating number is purely arbitrary and does not attempt to indicate the characteristics of the filler metal. A detailed basic system has been drawn up based on the former viewpoint; this system has been temporarily deferred to permit consideration of the latter viewpoint. A compromise system may result which will satisfy all present and anticipated requirements of a designation system for filler metal.

This report has been submitted to letter ballot of the committee which consists of 68 members; 50 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. H. DEPPELER,
Chairman.

S. A. GREENBERG,
Secretary.

REPORT OF JOINT RESEARCH COMMITTEE*

ON

EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS

PROGRESS REPORT TO THE SPONSOR SOCIETIES

During the past year, the Joint Research Committee on Effect of Temperature on the Properties of Metals of the American Society of Mechanical Engineers and the American Society for Testing Materials, has held two meetings. One was held on December 5, 1946, in New York, N. Y., during the annual meeting of The American Society of Mechanical Engineers. The second was held on May 7, 1947, at the headquarters of the American Society for Testing Materials in Philadelphia, Pa.

In addition, the committee sponsored three sessions at the annual meeting of The American Society of Mechanical Engineers, in New York; two on December 5 on the subject of Graphitization, and one on December 6 on the subject of Gas Turbine Materials. The following papers and reports were presented:

Graphitization:[1]

"Progress Report for Project No. 29 of the Joint A.S.T.M.-A.S.M.E. Research Committee on the Effect of Temperatures on the Properties of Metals. Studies on Susceptibility of Casting Steels to Graphitization," by J. J. Kanter and E. A. Sticha.

"Report on Graphitization Studies on High-Temperature Welded Piping of the Philadelphia Electric Company," by E. L. Hopping and A. E. White.

"Graphitization Studies of Materials for High-Temperature Service in Steam Plants," by W. G. Conant and W. A. Reich.

"Continuation of the Joint E.E.I.-A.E.I.C. Investigation on Graphitization of Piping," by S. L. Hoyt and A. M. Hall.

"Influence of Postweld Heat-Treatment on Graphitization," by I. A. Rohrig and Arthur McCutchan.

TABLE I.—SUMMARY OF RECEIPTS AND EXPENDITURES OF COMMITTEE FUNDS.

Balance as of June 30, 1946, shown in 1946 Report			$11 500.69
	RECEIPTS		
1946			
July	Engineering Foundation	$1 000.00	
September	Interest on funds (10/1/45 to 9/30/46)	75.18	
	Total Receipts		1 075.18
			$12 575.87
	EXPENDITURES		
1946			
August	Ogilvie Press for printing	$8.93	
December	Mack Printing Co. for 1945 Reports	46.70	
	Total Expenditures		55.63
Balance on hand, June 20, 1947			$12 520.24

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] *Transactions*, Am. Soc. Mechanical Engrs., 1947.

Gas Turbine Materials:[2]

"Nickel-Chromium Alloys for Gas-Turbine Service," by C. A. Crawford.

"Materials for Power Gas Turbines," by C. T. Evans, Jr.

"Precipitation-Hardened Alloys for Gas-Turbine Service," by Howard Scott and R. B. Gordon.

Finances:

There appears in Table I a summary of the administration of committee funds during the period from July 1, 1946, to June, 1947.

Organization and Membership:

There has been but one change in membership during the year. L. Schapiro, Chief Metallurgist, Douglas Aircraft Co., has replaced H. E. North, representing the aviation interest.

The committee has been considering the need for reorganization during the past year. At the May meeting it was agreed to reorganize on the basis of a central control or executive committee, appointed by the Sponsor Societies, and a number of sections to deal with needs in various fields of activity. For the present, sections are being organized as follows:

1. Steam power,
2. Gas turbines,
3. Petroleum and Chemical,
4. Aviation,
5. Nuclear Power (tentative),
6. General Research,
7. Data and Publications, and
8. Finances.

Research Projects:

Project No. 29 on Stability of Steels as Affected by Temperature (J. J. Kanter, chairman).—A report on this project prepared by E. A. Sticha, is appended hereto.

Respectfully submitted on behalf of the Joint Research Committee,

N. L. MOCHEL,
Chairman.

H. C. CROSS,
Secretary.

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[2] *Transactions,* Am. Soc. Mechanical Engrs. Vol. 69, No. 6, August, 1947.

APPENDIX

STABILITY OF STEELS AS AFFECTED BY TEMPERATURE[1]

(PROJECT NO. 29)

Project No. 29 of the Joint A.S.T.M.-A.S.M.E. Research Committee on Effect of Temperature on the Properties of Metals was established to investigate the "Stability of Steels as Affected by Temperature" and was particularly concerned with graphite formation near welded joints in high-temperature steam power piping. Two progress reports[2, 3] have been presented on that phase of the program which deals with castings, conducted under the sponsorship of the Manufacturers Standardization Society of the Valve and Fittings Industry. This work, embracing aging periods up to 10,000 hr. at 1025 F., has yielded certain pertinent information. The increase in graphitization susceptibility with increased aluminum additions for deoxidation was demonstrated conclusively. Retarding effect of normalizing after welding as well as of small alloying additions of chromium (up to ½ per cent) was shown. However, neither of these treatments when applied to material made by the usual foundry technique offered promise as means of preventing graphitization in steam service at 1000 F., in as much as aging for 10,000 hr. at 1025 F. produced graphite formation. Examination after 15,000-hr. aging has shown relatively little change from the 10,000-hr. results, so no further report on this part of the program will be presented here.

At the 10,000-hr. period it was apparent that steels of greater alloy content should be investigated also and, consequently, specimens of grade WC1 steel of A.S.T.M. Specifications A 217[4] plus 1 per cent of chromium, grade WC4 steel of Specifications A 217, and grade C3A steel of specifications A 157[5] were subjected to test. Conditions of test were exactly the same as in previous studies[2] except that aging was carried out at 1100 F. instead of at 1025 F., the latter change being instituted to accelerate the graphitization process if possible. While only meager results are available at this time, there is some question as to whether acceleration has resulted through use of the higher temperature. For aging times up to 6000 hr., however, none of these more highly alloyed steels has shown any graphitization.

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[1] This report was prepared by E. A. Sticha, Assistant Materials Research Engineer, Crane Co., Chicago, Ill., and submitted by J. J. Kanter, Chairman, Project No. 29, Materials Research Engineer, Crane Co., Chicago, Ill.

[2] J. J. Kanter, "Studies of Susceptibility of Casting Steels to Graphitization," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 68, pp. 581-587 (1946).

[3] J. J. Kanter and E. A. Sticha, "Studies of Susceptibility of Casting Steels to Graphitization," Appendix to Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 180 (1946).

[4] Tentative Specifications for Alloy-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 217 - 46 T), 1946 Book of A.S.T.M. Standards, Part I-A, p. 887.

[5] Standard Specifications for Alloy-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service (A 157 - 44), *Ibid.*, p. 274.

The Pipe Fabrication Institute agreed to cooperate with Project No. 29 also and offered, as its contribution, to study the effect of variables in welding procedure on the graphitization process in wrought material.

The following fabricators submitted samples for the program:

American Locomotive Co.
Crane Co.
Grinnel Co.
M. W. Kellogg Co.
Pittsburgh Piping & Equipment Co.

The committee chose A.S.T.M. Specifications A 280[6] seamless pipe for the study, and the Pipe Fabrication Inst. purchased from Pittsburgh Steel Co. a sufficient amount of schedule 160 material ($12\frac{3}{4}$-in. outside diameter by $1\frac{1}{4}$-in. wall thickness) of the following analysis and made by the open-hearth process:

Silicon, per cent	0.26
Manganese, per cent	0.60
Sulfur, per cent	0.020
Phosphorus, per cent	0.013
Carbon, per cent	0.19
Chromium, per cent	0.46
Molybdenum, per cent	0.46

Deoxidation was effected by use of $\frac{1}{2}$-lb. aluminum per ton of steel. Welding rod of the same nominal composition was employed in the form of $\frac{1}{8}$ and $\frac{5}{32}$-in. rods. Joint preparation and choice of welding procedure and rod size were left to the discretion of the cooperator.

Each fabricator received a length (10 ft. minimum) of annealed pipe which was cut in half and one section normalized from 1650 to 1700 F. after 2 hr. at temperature. The annealed and normalized pieces were then welded in a fixed horizontal position, no preheat being employed, and the joint designated as weld 1. After cutting 16 in. from the joined pipe with the weld at the center, the pieces were welded a second time, this time with a minimum preheat temperature of 500 F. The assembly was stress-relieved immediately at 1150 F. for 2 hr. after which a 16-in. length with the weld at the center was removed. In this condition the joint was designated as weld 2. This 16-in. section was split in half longitudinally, and one section stress-relieved at 1350 F. for 2 hr., the latter piece then being designated as weld 2A.

Once again the annealed and normalized pipe sections were welded with a minimum preheat temperature of 500 F., but this time the assembly was allowed to cool to room temperature before applying the stress-relieving treatment at 1150 F. (2 hr.), the joint in this state being designated as weld 3. As before, a 16-in. length with the weld at the center was cut from the assembly, split in half longitudinally, and one half stress-relieved at 1350 F. for 2 hr., the latter portion of the joint being marked weld 3A.

Realizing that susceptibility to graphite formation in A 280 pipe should be much lower than in carbon-molybdenum steel pipe, aging of the test specimens was carried out at 1100 F. with the hope that results might be obtained in a reasonable time. Two samples representing extremes of welding conditions, that is, the horizontal and overhead position, were taken from each of the five welds and submitted for test. Examination of structure was made after aging for 1500, 3000, 4500, and 6000 hr. but as yet no graphite has been detected in any of the specimens.

There has been some spheroidization of carbides in the heat-affected zone but not to any great extent. A fine precipitate was observed in the ferritic areas of most of the specimens, apparently in greater amounts in the annealed pipe. The annealed material exhibits a banded

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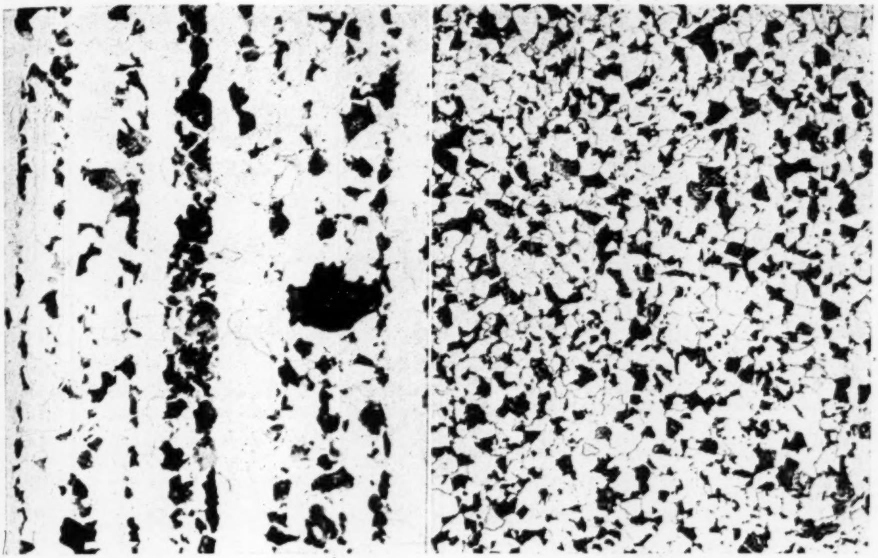
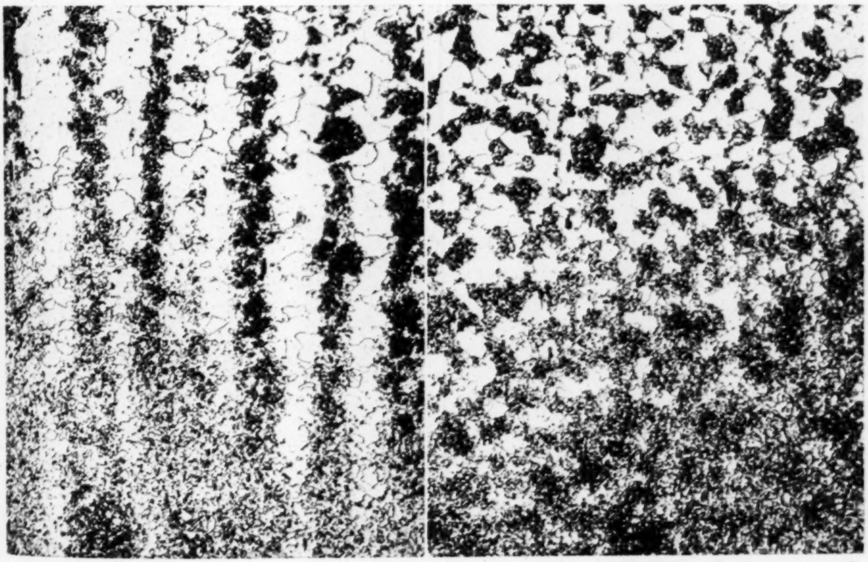
[6] Tentative Specifications for Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 280 – 46a T), *Ibid.*, p. 940.



(*a*) As received pipe, annealed (*b*) Normalized pipe



(*c*) "Contact" zone, annealed pipe, aged 1500 hr. at 1100 F. (*d*) "Contact" zone, normalized pipe, aged 1500 hr. at 1100 F.

FIG. 1.—Microstructure of ½ per cent Chromium, ½ per cent Molybdenum Seamless Steel Pipe.

structure due to the prolonged cooling through the critical range. This structure suggests the possibility of large differences in directional properties. Creep studies have been made at 1000 F. on specimens having their axes taken transversely as well as longitudinally. Creep strength in the transverse direction is only slightly less than in the longitudinal direction, but seems fully equivalent to the values which have been approved for applications of this composition.

REPORT OF ADVISORY COMMITTEE
ON
CORROSION

This is the first report presented by the Advisory Committee on Corrosion, as such, although the individual committees represented on the Advisory Committee have been reporting regularly each year.

Research activities in the field of corrosion have been carried on in the Society for the most part by the individual committees interested in specific materials. There was no formal coordination of this work other than by interlocking membership and cooperation between individual committees working in a field of mutual interest. Such was the case in the early cooperative tests carried out by Committee D-1 and Committee A-5, mentioned in this report.

In 1928 a more formal coordination of mutual interest was recognized in the formation of the Coordinating Committee on Corrosion composed of representatives of Committees A-5, A-10, B-3, and B-4. A representative of Committee B-6 was added in 1930 and the committee functioned until 1941 when it was felt that a more inclusive committee was needed and closer cooperation required than could be secured through the operation of the old committee.

Accordingly, a conference was held in June 1941, under the auspices of Committee E-9 on Research, of the representatives of Committees A-5, A-10, B-3, B-6, and B-8. As a result of this conference there was formed the present Advisory Committee on Corrosion. The Representatives of the committees mentioned immediately above and also of Committees B-4, B-7, D-1, and D-19 and three members-at-large constitute this committee.

Within the past two years the advisory committee has been assigned the responsibility for the acquisition and maintenance of test sites for use by all committees of the Society for atmospheric exposure testing. Activity along this line has been the principal concern of the committee during the past year. The Society is at present using some twenty-five different exposure sites, for most of which we have no clearly defined security of tenure. The committee plans to fix upon a smaller number of sites, adequately distributed so as to give representative conditions, and to arrange for definite rights for occupancy for a reasonable period of time. These sites will be suitable for exposure testing of paints, plastics, textiles, ceramics, and other materials as well as metals. Preliminary surveys of possible sites are now under way.

In 1946 there was added to the Society's Regulations Governing Technical Committees a section outlining the duties and responsibilities of the advisory committee and its relation to the work of other committees.

Officers of the committee elected for the next two years are as follows:

Chairman, C. D. Hocker.
First Vice-Chairman, F. L. LaQue.
Second Vice-Chairman, R. B. Mears.

Appended to this report is a summary, prepared by the secretary, of the corrosion work of all A.S.T.M. Committees from the beginning of the Society's activities in this field to date.

W. H. Finkeldey,
Chairman.

Carter S. Cole,
Secretary.

APPENDIX

SUMMARY OF WORK ON CORROSION BY A.S.T.M. COMMITTEES

The review of A.S.T.M. research projects on properties of materials and methods of testing published in the December, 1943 BULLETIN contains considerable data on corrosion tests completed, under way, or contemplated as of that date. The report which follows is an account of the corrosion work that has been undertaken by various committees and summarizes briefly the principal activities that each has followed. Unless otherwise indicated details will be found in committee reports or papers in the *Proceedings* for the year indicated. It should be noted that between the years 1914 and 1938 (except for 1921) the *Proceedings* were published in two volumes with the committee reports in Part I and papers in Part II. Up to and including 1913, in 1921, and from 1939 to date, committee reports and technical papers are in the same volume.

Corrosion testing and corrosion testing procedure and methods have been important subjects of study by A.S.T.M. committees from the very early days of the Society. These studies have made an important contribution to our knowledge of engineering materials and have been the basis for many specification requirements, particularly in the field of protective coatings, both metallic and non-metallic. The establishment of the copper requirements of copper-bearing steel for its effectiveness in resisting atmospheric corrosion is a direct result of A.S.T.M. corrosion studies and numerous other instances could be cited.

COMMITTEE A-5 ON CORROSION OF IRON AND STEEL

(Organized in 1906)

Earlier Work:

Committee A-5 is one of the oldest committees in the Society actively engaged in corrosion research. It was organized in 1906 as Committee U. In the *Proceedings* for that year papers by Snow, Toch, and Howe and their discussion outlined various problems for the committee to undertake. The first report of the committee was published in 1907 with additional papers by Cushman (chairman of the committee) and by Walker and Dill, while the 1908 report outlined more clearly the various problems before the committee and papers were presented by Cushman and by Howe and Stoughten.

In 1909 the committee reported 12 samples of galvanized wire with different coatings exposed at Pittsburgh. An interim report on these tests was given in 1910 (the committee then had its present designation A-5) and a complete report on the same tests in 1911. In the meantime the committee in cooperation with Committee D-1 reported in 1910 the results of tests on painted steel fence panels erected in 1908 at Atlantic City. In 1911 papers were presented in the "Sulfuric Acid Corrosion Test" by Chapman; "On the Marked Influence of Copper in Iron and Steel on the Acid Corrosion Test" by Walker; and the "Rate of Corrosion of Metals Exposed to Locomotive Gases" by Carpenter.

There was no report of committee activities in 1912, 1913 or 1914; the committee was reorganized in June, 1914.

Later Tests:

Atmospheric Corrosion Tests of Copper-Bearing and Non-Copper-Bearing Uncoated Sheets.—In 1915 the committee took the preliminary steps for testing the effect of steel composition by exposure tests at Fort Sheridan, Ill., Pittsburgh, Pa., and Annapolis, Md. Analysis of the sheets used are given in the 1916 and the 1918 reports and actual exposure started in 1916–1917. Cumulative reports on the tests at all three locations are given in 1918, 1920, 1921, 1922, and 1923 when the Pittsburgh tests were completed. Reports on the two remaining locations continued in 1924, 1925, 1926, 1927, and 1928, when data on the Fort Sheridan tests were presented in detail as these tests had to be concluded because the space used was no longer available. The Annapolis reports have continued to date with interim or detailed reports presented yearly. The 1946 report of the committee gives cumulative results of the tests at all three test sites.

Zinc-Coated Sheets.—In 1917 the committee also published a report on preservative metallic coatings for metals which discussed methods of testing zinc coatings in great detail. In 1918 there was a paper by Rawdon on "Structure of Zinc Coatings," and by Finn on "Salt Spray Test Methods." Further notes on the testing of zinc coatings are given in the 1919 report.

Total Immersion Test.—Total immersion tests were started by the committee in 1920 and results of immersion in mine water at Calumet, Pa., reported in detail in 1921. Additional reports on the same tests in tap water at the National Bureau of Standards and in Severn River water at Annapolis are given in 1924. More detailed reports were given in 1925 and in 1926 and a full report on sheet specimens in 1927. Additional test locations were added from time to time and more detail of the results of these tests is given yearly up until 1936. The tests on the pipe specimens were concluded in 1940, but stress of more important activities during the war has so far prevented the final completion of a report thereon.

Sulfuric Acid Tests.—Tests started in 1930, and reported in detail in 1931 were undertaken to determine if correlation could be established between the 20 per cent sulfuric acid test (the pros and cons of which had been argued since the committee was formed in 1906) and the results obtained on the same types of material in long-time atmospheric corrosion testing. The committee found that the sulfuric acid test was not capable of consistent repetition nor of correlation with service and that data from such tests should not be used to forecast the relative life of ordinary ferrous materials in the atmosphere.

Atmospheric Corrosion Tests—New Series:

In 1920 the committee recognized the desirability of undertaking a very extensive corrosion test program and plans for these tests materalized during the next few years while the funds necessary for carrying them out were obtained by special subscription. The tests, as finally organized, divided into three distinct groups and reports on progress to date are given below.

Atmospheric Corrosion Tests of Uncoated and Galvanized Corrugated Sheets.—Committee A-5 began studies in 1926 at five test locations (Altoona, Pa.; Brunot Island, Pittsburgh, Pa.; Sandy Hook, N. J.; State College, Pa.; Key West, Fla.) representing various atmospheric conditions, on corrugated sheets, both uncoated and galvanized with various weights of zinc coatings. A report of failures of all galvanized sheets after 7-yr. exposure at Altoona and of progress at other sites was published in 1934, and failure of all sheets after $8\frac{1}{2}$-yr. exposure at Pittsburgh in 1935. A brief report of correlation of results of the Brunot Island tests on sheets, hardware, and structural shapes and electroplated coatings on steel was included in the 1936 report.

In 1938 initial failures observed at State College were reported; a comparison was made of the relative severity of the five locations on coated and uncoated sheets; the behavior of coated sheets at five test locations was described. The 1944 report records (*1*) coating failures at Sandy Hook and State College; (*2*) the behavior of coatings at Key West in detail; (*3*) ten uncoated sheets exposed at five test locations to show contribution of coatings to life of sheets; (*4*) data on time elapsing between exposure and perforation. Loss of weight experiment at Sandy Hook involving seasonal variations started April 1939. Interesting details of this test and further data on specimens at State College and Key West were reported in 1942. Later data on the galvanized sheet exposure tests are given in 1946, and further data will be recorded in 1948.

Atmospheric Corrosion Tests of Metallic-Coated Hardware, Structural Shapes, Tubular Goods, etc.—In 1928 Committee A-5 began studies at the same five test locations noted above, representing various atmospheric conditions, on metal products coated with the following eight types of coatings by commercial operations: Hot-dipped zinc, electrodeposited zinc, sherardized (zinc) applied in gas-heated drum, sherardized (zinc) applied in electrically heated drum, electrodeposited cadmium, hot-dipped aluminum, hot-dipped lead (Amaloy), parkerized. Diagrams showing the rack mountings of the various specimens are given in the 1929 report. Results of inspections of the specimens at all test locations after 2-, 4-, and 6-yr. exposures were reported in 1931, 1933, and 1935 respectively. In 1936 a brief report was given on correlation of results of this project with other corrosion studies by the committee. In 1938 extensive data were given showing rusting that has occurred on samples since the 1935 report.

Later the hardware racks at State College and Sandy Hook were reconditioned with numerous specimens remounted—largely those with hot-dip zinc, aluminum or lead coatings and some sherardized coatings, and the 1943 report shows the identification and positioning on the racks of the remounted samples as well as the positions on the original racks. A full report on the progress to date is contained in the 1944 *Proceedings*, where it is noted that the series of tests at Pittsburgh and Altoona were involuntarily concluded due to loss of all specimens in 1939 and 1942 respectively. Data on results at the three remaining locations is given in the 1946 report. Additional hardware tests are now being planned (1947).

Atmospheric Corrosion Tests on Wire and Wire Products.—Extensive country-wide outdoor tests of wire, and wire products, including unfabricated wire, barbed wire, wire strand, farm-field fencing and chain-link fence as outlined in the 1934 report were started in 1936 at 11 test locations (Sandy Hook, N. J.; Bridgeport, Conn.; State College, Pa.; Lafayette, Ind.; Ames, Iowa; Manhattan, Kans.; Ithaca, N. Y.; Santa Cruz, Calif.; College Station, Texas; Davis, Calif.; Brunot Island, Pittsburgh, Pa.). Materials include uncoated wire and fencing (both copper-bearing and non-copper-bearing), zinc-coated wire and fencing, corrosion-resistant steel wire and fencing, and lead-coated wire and fencing. Test sites are representative of atmospheric conditions from coast to coast.

The 1937 committee report contains detailed data on the installation of test materials. In the 1939 report there were given the results of 2-yr. exposure tests at all locations and detailed data of referee tests at the National Bureau of Standards to provide complete information on original characteristics of the materials on test, including photomicrographs, etc.

The 1943 report gives results of tests after 6-yr. exposure. The 1945 report gives 8-yr. results. At each location, there are more than 900 specimens including short lengths of wire and wire strand at all eleven locations, farmfield fence at nine sites, barbed wire and chain-link fence at eight test sites. The latest full report on these tests is in the 1947 report of the committee, and covers both the 1945 and 1946 inspections.

Other Tests:

Accelerated Laboratory Tests.—The committee has been interested in accelerated laboratory testing, as well as outdoor exposure, and papers on salt spray testing will be found as far back as 1914 (Kapp) and 1918 (Finn). Later work on salt spray has been in cooperation with Committee B-3 and the present status of the work will be found under the report of the work of that committee. Various accelerated laboratory tests were also reported in 1924 by Krynitski, Rawdon, and Finkeldey.

Embrittlement Test.—Work on embrittlement testing started in 1931 with an extended discussion and bibliography of the subject followed by a paper by Epstein in 1932. A recommended practice for the embrittlement test (A 143) was published as tentative in 1932 and advanced to standard in 1935.

Electrodeposited Coatings.—A special subcommittee, working jointly with the American Electroplaters' Society and the National Bureau of Standards, was organized to study this subject in 1931 and exposed test specimens in 1932 at six locations and data were reported in 1933, 1934 and 1936. See also Research Paper No. 867 of the National Bureau of Standards (Vol. 16, No. 2, 1936).

Methods of Test for Coatings.—Extensive studies involving tests for determining weight and uniformity of zinc coatings on hardware and other shapes; and methods for plated coatings including metallographic, stripping, dropping, spot tests, etc., resulted in various tentative standards. These cover the following: Weight of Coating on Zinc-coated Iron and Steel Articles (A 90), Uniformity of Coatings by Preece Test (copper sulfate dip) on Zinc-coated (galvanized) Iron or Steel Articles (A 239), Local Thickness of Electrodeposited Coatings (A 219), and Weight and Composition of Coating on Long Terne Sheets by the Triple Spot Test (A 309).

A special group issued a report (March 1942 *Bulletin*) on the demand for, and relative merits of, methods for the dropping test to determine thickness of zinc and cadmium coatings on steel. Some consideration was also given by this group to magnetic test methods for determining the thickness of coatings.

Methods of Testing.—A subcommittee on this subject is currently (1947) investigating the limits of accuracy of the various field testing methods (such as dropping test methods and magnetic test methods) now employed to determine the thickness of protective coatings.

Specifications.—Ten Specifications for zinc coatings (galvanizing) of various iron and steel products and articles have been developed by the committee. These include sheet (A 93), line wire (A 111), tie wire (A 112), farm fencing (A 116), chain link fence (A 117), barbed wire (A 121), wire strand (A 122 and A 218), structural shapes (A 123) and hardware (A 153). Other specifications, including electrodeposited coatings (A 164) (zinc) and (A 165) (cadmium), also lead coatings (A 267), have been written. Work on specifications for long terne sheets was completed in 1947 and issued (A 308).

Joint Symposiums.—In addition to the data reported above, attention is also directed to the separately published (Technical Publication No. 17) Symposium on the Outdoor Weathering of Metals and Metallic Coatings. This symposium was jointly sponsored by Committees A-5 and B-3 in 1934 and contains papers by C. D. Hocker, R. F. Passano, G. N. Schramm and E. S. Taylerson, W. H. Finkeldey and C. L. Hippensteel. Another Symposium on Corrosion Testing Procedures was jointly sponsored by these same committees in 1937 (see under Committee B-3 below).

COMMITTEE A-10 ON IRON-CHROMIUM, IRON-CHROMIUM NICKEL, AND RELATED ALLOYS

(Organized in 1929)

As noted later in this report under the heading of Committee B-4, the symposium sponsored by Committee B-2 in 1924 led indirectly to the establishment of Committee A-10. Attached to this symposium in the 1924 *Proceedings* was an extensive tabulation of the properties of corrosion-resistant and heat-resistant alloys. Responsibility for this tabulation was taken over by Committee A-10 and a greatly enlarged presentation of data was published by Committee A-10 in the 1930 *Proceedings.* Subsequently, additional information has been added from time to time and the information has been separately published as Tables of Data on Corrosion Resisting Steel. As this report is published, the latest edition of the data is 1942 and the information covers wrought steels only. Data on cast steels has been assembled by the committee and will be combined with the earlier information for separate publication within the next year.

The corrosion testing work of Committee A-10 began with its organization in 1929 under the direction of its Subcommittee on Methods of Corrosion Testing, which published its first report in 1933. This report included analyses of salt-spray testing, copper sulfate stain testing, and boiling nitric acid testing.

In 1934 further consideration was given to these subjects, but no work was published. The committee cooperated with various other A.S.T.M. committees on corrosion and related problems. In 1938 a committee was appointed to examine architectural installations of stainless steel, and in 1939 its report was published appended to the committee report. Appearing also was a "Guide for Plant Corrosion Tests" which was revised and published in 1940 as "Tentative Recommended Practice for Conducting Plant Corrosion Tests" (A 224); first issued in 1944, this was revised in cooperation with Committee B-3 in 1946 to make the method applicable also to non-ferrous metals.

A supplementry Report on Inspection of Stainless Steel Structures was published in 1940, and the committee began working with Committee B-3 on studies of atmospheric galvanic corrosion of stainless steel in contact with nonferrous metals and alloys.

In 1941 work was begun on a general procedure for testing in boiling liquids. In 1942 this work resulted in a Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (A

262); in 1943 and 1944 revisions were made in this Practice. In 1943 a Proposed Tentative Method of Total Immersion Testing of Stainless Steels (A 279) was formulated, and was revised for publication in 1944.

In 1944 an initial outline of a program for Atmosperhic Corrosion Testing was set up and was divided among seven subgroups; formulation of this program is now nearing completion. The committee also prepared for Frankford Arsenal a summary on the Passivation of Stainless Steels, which is appended to the 1946 committee report, as well as data on the performance of corrosion-resistant steel on the deck houses of two Navy destroyers.

The committee has participated actively in programs with other committees of A.S.T.M. on problems pertaining to corrosion. The committee proposes to carry out such additional investigations as may be required from time to time to enable precise interpretation of results of tests carried out in accordance with A.S.T.M. recommended practices. Consideration will be given to recent proposals that the committee develop a recommended practice for galvanic corrosion tests, preferably in cooperation with other corrosion committees. Similarly, the committee will work with other corrosion committees toward the development of other corrosion testing methods that are generally applicable to a variety of metals and alloys.

The Subcommittee on Methods of Corrosion Testing arranged for a symposium on Atmospheric Weathering of Corrosion-Resistant Steels held at the A.S.T.M. Annual Meeting in June, 1946, which consisted of seven papers and outlined past experience with corrosion-resisting steels. (This symposium is published in the 1946 *Proceedings* and also separately.) The subcommittee also has completed details of a plan for a new 15-yr. exposure test of corrosion-resisting steels which, after final review by members of the entire committee, will be started in the near future.

COMMITTEE B-3 ON CORROSION OF NON-FERROUS METALS AND ALLOYS

(Organized in 1922)

Committee B-3 was organized in 1922 for the purpose of improving the technique of corrosion testing in the field of non-ferrous metals, of standardizing it so far as possible, and later of assembling standard data on the corrosion resistance of different non-ferrous metals and alloys.

Completed and Current Work:

As the first project of the committee, cooperative corrosion tests were conducted at 14 laboratories on a series of companion samples of 6 non-ferrous metals, by 4 methods—simple immersion, alternate immersion, spray, and accelerated electrolytic. Six solutions were used in each case, 2 acids, 2 alkalies, 2 neutral solutions. The correlated results, published in 1927 have served as background for the later standardization of these corrosion test procedures.

Plans maturing over a period of several years for the study of corrosion of non-ferrous metals under different conditions of service culminated in 1931 in the starting of three series of tests. These included long-time outdoor exposure tests; corrosion in liquids (manufacturers' plant conditions); and galvanic corrosion of coupled metals under outdoor exposure conditions.

Outdoor exposure tests.—At nine widely separated sites in various parts of the United States (State College, Pa.; Phoenix, Ariz.; Pittsburgh, Pa.; Altoona, Pa.; Rochester, N. Y.; New York City; Sandy Hook, N. J.; Key West, Fla.; La

Jolla, Calif.), exposure tests of projected 20 to 25 years' duration were started in 1931 on 24 representative non-ferrous metals and alloys. The sites were typical of heavy industrial, light industrial, rural, seacoastal, and inland dry atmospheres. Specimens were of 2 types, precut tension test specimens and 9 by 12-in. sheet (see 1932 report). These were to be used for measuring the corrosion rate by progressive change in tensile properties and also by loss of weight and change in surface characteristics. Samples were withdrawn at intervals for testing.

The first phase of the work (10-yr. period) was finished in 1941, the results being published in the committee's reports for 1943 and 1944. These reports formed the basis for a series of six papers which constituted a separately published symposium (Technical Publication No. 67) on Atmospheric Exposure Tests on Non-Ferrous Metals at the 1946 Spring Meeting of the Society. Specimens still remaining on the exposure racks constitute the second phase of the long-time outdoor exposure tests and will be allowed to remain there undisturbed for another ten years before the test is terminated in 1951.

Corrosion in liquids.—This series of tests was planned to be representative of corrosion conditions in manufacturers' plants in a number of common chemical solutions. Many of the materials in these tests were identical with those of outdoor exposure tests. Because of the unsettled industrial conditions during the depression, the progress of this series was disturbed considerably. However, the tests were completed and the results published in 1934, 1935, and 1938.

Galvanic electrolytic corrosion in atmospheric exposure.—Materials used were those of the outdoor exposure tests and typical 2-metal couples were exposed in 1931 at the same test sites. The final report was published in 1939. Supplementing this series another series was started in 1941, with stainless steel specimens (2 types—304 and 316) coupled to eight selected non-ferrous metals and to mild steel. Three of the exposure sites of the previous series (New York City, State College, and Altoona) are being utilized, together with a new one at Kure Beach, N. C. (see 1942 report). The first set of specimens was removed in 1946 at the end of five years' exposure, tested for contact resistance, disassembled, and visually inspected. The data will be reported when weight changes have been determined.

Standard Corrosion Procedures.—The early work described formed the background for a symposium sponsored by Committees A-5 and B-3 in 1937. The complete report (7 papers and discussion) has been published by the Society under the title, "Symposium on Corrosion Testing Procedures" (Technical Publication No. 32). It has also culminated in the adoption by the Society of standard procedures for total immersion corrosion test (B 185); alternate immersion corrosion test (B 192); and salt spray corrosion (fog) test (B 117). Committees A-5 and D-1 cooperated in developing B 117. Further study of salt spray procedure continues and cooperative tests to determine whether B 185 will give consistent results are being undertaken.

Proposed Future Work:

Long-time exposure tests of newly developed non-ferrous alloys are under consideration with special attention being given to aluminum alloys and magnesium alloys for use by the aircraft industry. The tests will be conducted along the same lines as tests of corrosion in the atmosphere as reported above.

Galvanic Electrolytic Corrosion.—A

subcommittee is actively engaged in preparing electrolytic couples for the study of magnesium when coupled to other common metals. Plans were perfected for obtaining all the various metals with the help of a number of manufacturers, and it is anticipated that the assembled couples will be placed during the summer of 1947 for exposure at four typical locations. Efforts are being made to obtain one site representative of tropical climate. The work necessitates extensive cooperation of Committee B-7 on Light Metals and Alloys. (See 1946 report of Committee B-3 for details.)

Weather.—A subcommittee has been formed to study the various elements which make up "weather" and to attempt a "more precise evaluation of those weather factors which influence atmospheric corrosion and a correlation of such factors with the performance of materials under test." The ultimate aim would be to extend the basis for the intelligent application of results of atmospheric exposure tests, particularly in respect to their use in estimating performance of materials at locations other than those at which large scale tests are made. (See 1943, 1946, and 1947 reports.)

Salt Spray.—The salt spray test has continued to be extensively used by industry, and a subcommittee is now engaged in determining the factors essential in this test method so that complete uniformity and consistency in testing by various testing engineers will be possible.

Other Activities.—The subcommittee on Total Immersion Corrosion Tests approved in 1946 changes suggested by a subgroup which would make the Recommended Practice on Plant Corrosion Tests (A 224), developed originally for corrosion-resisting alloys, applicable to the testing of non-ferrous metals.

The Subcommittee on Statistical Analysis and Planning of Corrosion Testing will continue to aid other subcommittees in planning tests so that data obtained may be treated statistically and will consider the preparation of a simple outline procedure for applying statistics to corrosion tests.

Humidity.—A new Subcommittee on Humidity was organized in February, 1947, and will study the present status of humidity testing and develop standard methods for this important subject. During the war years, corrosion resulting from humidity in the tropics was excessive and troublesome. Certain industrial applications relate to refrigerators, air conditioning and building insulation.

COMMITTEE B-4 ON ELECTRICAL HEATING, RESISTANCE, AND RELATED ALLOYS

(Organized in 1925)

A Symposium on Corrosion-Resistant, Heat-Resistant and Electrical-Resistant Alloys, suggested by Committee B-2 on Non-Ferrous Metals, was held in 1924 (see papers in 1924 *Proceedings*). This led directly to the establishment of Committee B-4 in the following year (and indirectly, several years later on the recommendation of a joint committee of representatives of Committees A-5 and B-3 in 1928, to the establishment of Committee A-10).

The interest of Committee B-4 in corrosion testing has been primarily in the field of high temperature service and in 1937 a Subcommittee on Methods of Test for Alloys in Controlled Atmospheres was organized. This committee studied the effect of furnace atmospheres on resistors and evolved a method of test which was tried out in four cooperating laboratories. This resulted in the publishing of a Method of Test for the Effect of Controlled Atmospheres on

Alloys in Electric Furnaces (B 181). This method was published first in 1942 as information and then in 1943 as tentative. Work on similar tests on castings used in furnaces began in 1942 and continues, with a new test program being formulated to study the causes and prevention of "green rot" in electric furnaces using controlled atmospheres. A study of thermocouple protection tubes and their penetration by hydrogen has also been initiated.

COMMITTEE B-6 ON DIE-CAST METALS AND ALLOYS

(Organized in 1930)

Before Committee B-6 was organized, work in the die casting field was carried on by Subcommittee XV of Committee B-2. This subcommittee began its work in 1928, and in 1929 started atmospheric exposure in tests of about 35,000 test specimens of 21 different zinc-base and aluminum-base die casting alloys secured from 8 producers. Outdoor exposure locations were at State College, Pa.; Key West, Fla.; Sandy Hook, N. J.; Rochester, N. Y.; Altoona, Pa.; and New York City. Indoor exposure test locations were at Coco Solo, Panama Canal Zone; Hanover, N. Mex.; New Kensington, Pa.; and Cambridge, Mass. The 1929 report of Committee B-2 gives a statistical basis for analysis of the test results. A picture of one of the outdoor exposure test racks is shown in the 1930 report of Committee B-2. Committee B-6 was organized in June 1930, and subsequent data on the exposure tests are to be found in its reports.

Report in 1932 on one-year exposure tests on all locations except State College showed the definite superiority of Alloy XXI to the other experimental zinc base alloys, especially as indicated by the retention of impact strength. The results of these exposure tests led directly to the establishment of the specification for Zinc-Base Alloy Die Castings (B 86).

In 1935 the report of the committee gives the detailed results of the five-year exposure results on 12 aluminum and 10 zinc-base alloys, while the 1940 report gives further information on the five-year test as well as the physical properties and the expansion data after 10 years atmospheric exposure.

In 1945 the committee reported that all of the zinc and aluminum-base alloy test bars exposed in 1929 and the five-year test bars of zinc and magnesium alloys (1939 series) had been recalled. Data on these is in the 1946 Report. See also 1946 paper by Gohn and Menges. In addition two test specimens of special purity aluminum and one standard aluminum alloy from three outdoor locations after 10 years exposure have been recalled for examination. Data on these tests are also appended to the 1946 report. Future corrosion testing activities of the committee will be determined when the committee has had an opportunity to study these reports and so determine where further information is needed.

The data on its various tests published by the committee have been quite complete and some pertinent data have been added in each yearly report. In addition to those years which have been mentioned, see particularly 1932, 1934, 1937 and 1939.

The committee has also sponsored a number of papers on the subject of the die-casting alloys. Among these are the papers on the Effect of Variations in Aluminum Content on the Strength and Permanence of A.S.T.M. No. XXIII Zinc Casting Alloys by Anderson and Werley appended to the 1934 report; papers by Fox on Finishing of Die Castings (1936), by Werley on Improvement of the Soundness and Uniformity of Test Bars (1937), and by Winston on Magnesium Alloy Die Castings (1939), and Kelton on Fatigue Testing of Zinc-Base Alloy Die Castings (1942).

COMMITTEE B-7 ON LIGHT METALS AND ALLOYS, CAST AND WROUGHT

(Organized in 1928)

Tests of Aluminum and Aluminum Alloys:

Committee B-7 organized a Subcommittee on Anodic Oxidation of Aluminum and Aluminum Alloys in 1936. The test methods studied were (*a*) thickness, (*b*) abrasion resistance, (*c*) electrical breakdown voltage, of anodic coatings on aluminum. A discussion of the problem is given in an appendix to the 1937 report. In 1940, four papers dealing with various phases of the problem were presented by Messrs. Keller, Edwards, Arlt, and by Compton and Mendizza. The work of the committee has resulted in the publication of three test methods as follows: Dielectric Strength of Anodized Aluminum (B 110); Sealing of Anodically Coated Aluminum (B 136); Weight of Coating on Anodically Coated Aluminum (B 137). A new non-destructive method of test for anodic coatings by an electronic thickness gage was called to the attention of the committee and has been carefully studied. A report on this is being prepared.

Appended to the 1943 and 1945 reports are interesting comparisons of the Performance of Anodic Coatings on Wrought Aluminum Alloys When Exposed to the Salt Spray (B 117) and to the Weather. The salt spray testing was carried out at four laboratories. Atmospheric exposures (started in 1941) were made at Philadelphia; Chicago; New Kensington; Miami; Point Judith, R. I.; Oakland, Calif.; and New York City. The results, generally speaking, indicate that while salt spray exposure tests may discover coatings subject to early failure, still the resistance of most anodic coatings to this type of exposure is sufficiently extended so that the method could scarcely be considered an accelerated test.

Tests of Magnesium and Magnesium Alloys:

The committee prepared preliminary plans several years ago for a test to determine the corrosion behavior of the standard cast and wrought magnesium alloys but was prevented by the war from carrying out the program. The plans for a magnesium alloy test program have now been abandoned in favor of a more extensive investigation of both aluminum and magnesium alloys, to be conducted jointly by Committees B-7 and B-3. A questionnaire to develop the desires of the committee members has been circulated and the answers will be used as a basis for the program which, it is hoped, can be started during 1947.

COMMITTEE B-8 ON ELECTRODEPOSITED METALLIC COATINGS

(Organized in 1941)

Corrosion work in Committee B-8 started shortly after the organization of the committee in 1941, with the committe's investigation of current practices of salt spray corrosion testing of electrodeposited coatings.

Completed work of the committee consists of the summation and application of completed intersociety exposure tests in a series of specifications for electrodeposited coatings; and Subcommittee III's report (1943) on Salt Spray Corrosion Testing. Partially as a result of the latter, further A.S.T.M. effort has been directed toward improving the procedure of salt spray corrosion testing. This has finally resulted in the much improved B 117 – 44 T.

Lead Coatings.—Outdoor atmospheric exposure tests of various electrodeposited lead coatings on steel were started in 1944. The exposure sites for these tests are New York City, State College, Pa., Kure Beach, N. C., and Tela, Honduras. The test was described in detail in the 1944 report and results of the first two years exposure are given in the committee's report for 1946, and further data in the 1947 report.

Copper-Nickel-Chromium Coatings.—Outdoor atmospheric exposure studies of copper - nickel - chromium coatings on high-carbon steel of bumper bar grade were started in 1946. Test panels were exposed at New York, Pittsburgh, State College, Kure Beach, N. C. The purpose of the test is to determine the optimum ratio of copper to nickel in composite coatings of copper-nickel-chromium on high-carbon steel. Results of exploratory tests are reported in 1947 and will form the basis for a more extensive investigation.

It is planned to make studies of the effect of abrasive grain size used in polishing the base metal on the protective value of the electrodeposited coatings. Some work along these lines has already been done on steel by the American Electroplaters' Society, using A.S.T.M. racks for the exposure tests. Some of the work on steel will probably be repeated and the program expanded to include other base metals such as zinc and brass.

A study of the mechanism of corrosion through discontinuities in electrodeposited coatings will also be made. In this connection the committee hopes to promote fundamental studies of the problem, obtain data for the construction of polarization curves, observe and measure galvanic effects, etc.

It is also planned to develop and standardize accelerated corrosion tests for electrodeposited coatings which will be more suitable than those presently available. This would include studies of porosity testing methods with the view toward preparing Recommended Practices for the porosity testing of specific base metal-coating combinations.

Also planned are indoor exposure tests of electrodeposited coatings. There may also be an expansion of the current outdoor exposure testing program to include a broad range of electrodeposited coating - base metal combinations including alloy coatings, either electrodeposited as such, or subsequently alloyed by heat treating.

A subcommittee, which is concerned with Supplementary Protective Finishes for Metallic Coatings, discussed plans in 1946 and 1947 for evaluating various supplementary coatings, such as chromate and phosphate types on zinc and cadmium surfaces and appointed a subgroup to collect available data on the subject and to prepare a suitable program of tests.

COMMITTEE D-1 ON PAINT, VARNISH, LACQUER AND RELATED PRODUCTS

(Organized in 1902)

Committee D-1 was organized in 1902 as Committee E on Preservative Coatings for Iron and Steel and early considered field service and laboratory testing. Various subcommittees were organized immediately, and in 1906 an extensive test of paints on the Pennsylvania Railroad Bridge at Havre de Grace was started. The inspection methods for these tests are outlined in the 1907 report, and an analysis of the paints used is given in the 1908 report. Detailed reports on the results of the Havre de Grace tests were published in 1910, 1911, 1913, 1914 and 1915. In the meantime wooden panel tests had been started in Atlantic City in 1909 and reported on in 1910. An exposure test of steel panels made in cooperation with Committee A-5 was reported in 1911 and again in 1913, 1914, and 1915.

In 1916 a subcommittee was organized to study the preparation of iron and steel surfaces for painting. Test panels were prepared and exposed at Altoona and Brooklyn in 1917. Detailed reports of these tests were given in the 1918, 1919, 1921, 1923, 1924, 1925, 1926, and 1928 reports of the committee.

A study of chemical pretreatment of various steels, with subsequent painting and exposure, was started in 1939, reported on in some detail in 1941, and the first program of tests was completed in 1944.

White paints were exposed for test at Arlington, Va., in 1912 and reported on in detail in 1914. Tests of anti-fouling paints were started in 1921 at Norfolk, Va., Raritan, N. J., and Charleston, S. C., and reported in detail in 1922; again in 1923, 1924 and 1925. Results of exposure tests on varnish were also reported in 1923. In 1937 the committee sponsored a symposium on the Correlation Between Accelerated Laboratory Tests and Service Tests on Protective and Decorative Coatings. This is separately published as Technical Publication No. 33. In 1940 the committee published a report on the Comparison of Accelerated Weathering *versus* Outdoor Exposure for Paints, and further comparisons are contained in the 1942 report.

Committee D-1 cooperated with Committees A-5 and B-3 in the development of the Methods for Salt Spray Testing (B 117), and the results of the committee's work have resulted in the development of a number of specifications and methods of test, among which the following are typical:

Specifications for Wood to be Used as Panels in Weathering Tests of Paints and Varnishes, (D 358 - 44 T),

Method for Preparation of Steel Panels for Exposure Tests of Enamels for Exterior Service (Preparation of Steel Panels for Painting) (D 609 - 46 T),

Method of Evaluating the Degree of Resistance to Rusting Obtained With Paint on Iron or Steel Surfaces (Rusting) (D 610 - 43),

Method of Evaluating the Degree of Blistering of Organic Coatings on Metal when Subjected to Immersion or Other Tests Involving Exposure to Moisture or Liquids (Blistering) (D 714 - 45),

Method of Evaluating Degree of Resistance to Flaking (Scaling) of Exterior Paints of the Linseed Oil Type (D 772 - 47),

Method of Testing for Changes in Protective Properties of Organic Coatings on Steel Surfaces When Subjected to Immersion (Immersion Testing) (D 870 - 46 T), and

Recommended Practice for Characteristics of Standard Carbon Arc Accelerated Weathering Unit (Accelerated Weathering Unit) (E 42 - 42 T).

Current activities consist of further study of several methods of chemical pretreatment of steels, followed by painting and exposure; correlation of operating procedures for light and water exposure units; development of methods for humidity test, with and without condensation; study of methods for preparation of steel panels for painting; and accelerated tests of traffic paints. Work is proceeding satisfactorily on all of them.

The Subcommittee on Accelerated Tests for Protective Coatings has been especially active during 1946 and 1947, holding a number of meetings. New sections have been appointed on humidity testing and on alternate immersion and exposure conditions. The subcommittee is also conducting cooperative work on accelerated testing with the Cleveland Paint and Varnish Production Club.

Other proposed future work includes methods of preparation of non-ferrous metal surfaces for painting; and corrosion tests on steel structures in various locations, including evaluation of several methods of cleaning, to study the effect of variations in climate.

COMMITTEE D-19 ON WATER FOR INDUSTRIAL USES

(Organized in 1932)

A.S.T.M. interest in the work of the Joint Research Committee on Boiler Feed Water Studies led to the formation, in 1932, of Committee D-19. The committee, working with the Joint Research Committee, has studied various phases of water corrosion, particularly those relating to embrittlement, and this has led to the publication of an A.S.T.M. tentative method of Field Test for Tendency of Boiler Water to Cause Embrittlement Cracking of Steel (D 807).

This embrittlement tester is a design worked out by the committee, a previously suggested type having been described in a paper in 1938, by Straub and Bradburg, "A Method of Embrittlement Testing of Boiler Waters." Numerous other papers on corrosion testing of water have been published under the sponsorship of Committee D-19 including papers by Schroeder, Berk, and Partridge in 1936 and by Tajc in 1937. Other papers reporting phases of the same study have been published elsewhere and have a bearing on this subject. These include:

W. C. Schroeder and A. A. Berk, *Bulletin 443*, U. S. Bureau of Mines, 1941.

Schroeder and Berk and Stoddard, *Power Plant Engineering*, Vol. 45, August, 1941, pp. 76–79.

"Embrittlement Symposium," *Transactions*, Am. Soc. Mech. Engrs. Vol. 64, pp. 393–444 (1942).

S. F. Whirl and T. E. Purcell, *Proceedings*, Third Annual Water Conference, Engrs. Soc. of Western Penna., pp. 45–60 (1942).

Berk and Schroeder, *Transactions*, Am. Soc. Mech. Engrs. Vol. 65, pp. 701–711 (1943).

Another phase of corrosion study in Committee D-19 has been in connection with the work of the National District Heating Association. A paper on the N.D.H.A. Method of Measuring Corrosion by J. H. Walker was published in the 1940 *Proceedings* as part of a Symposium on Water. A Recommended Practice for the Use of N.D.H.A. Corrosion Tester (D 935) was completed in 1947. The Handbook on Corrosion of the Electrochemical Society contains a chapter on Boiler Corrosion written by the chairman and three other members of Committee D-19 and submitted on behalf of the committee. A Manual on Industrial Waters is being written by the committee.

MISCELLANEOUS

Not directly connected with the work of any particular committee was the Symposium on Protecting Metals Against Corrosion presented in Detroit in 1938 and separately published as Technical Publication No. 36. This includes papers by Gillett, Heussner, Wirshing, and McCloud.

REPORT OF COMMITTEE C-1*

ON

CEMENT

Committee C-1 on Cement held two well-attended meetings during the past year: one in December, 1946, at the Public Roads Administration laboratories near Washington, D. C.; the other in March, 1947, at Cincinnati, Ohio.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1946 Annual Meeting, Committee C-1 presented to the Society through the Administrative Committee on Standards a tentative revision of the Standard Method of Test for Compressive Strength of Hydraulic Cement Mortars (C 109 – 44). This tentative revision was accepted by the Standards Committee on September 9, 1946, and appears in the 1946 Book of A.S.T.M. Standards, Part II, p. 1701.

The committee records with sorrow the loss of two of its members by death, Guy W. Jordan and A. G. Fleming.

The Sponsoring Committee on Portland Cement (C. E. Wuerpel, chairman) has been studying numerous questions that have arisen concerning the specification requirements for portland cement. Those studies have led to certain proposals, presented elsewhere in this report, affecting the specifications for portland cement (C 150) and air-entraining portland cement (C 175). The sponsoring committee is continuing its studies of current problems, among them the question of including types III, IV, and V cements under Specifications C 175, and the question as to the adequacy of the air-entrainment limit now contained in that specification. The lower limit for air content has been considered as too low by some investigators, and the subcommittee has approved the proposal that the limits be changed from "16 ± 4 per cent" to "18 ± 3 per cent."

The Sponsoring Committee on Masonry Cement (H. D. Baylor, chairman) has started an investigation designed to secure data that would assist in writing a durability specification for masonry cement. Eight laboratories agreed to participate in that work, in which eight representative masonry cements are to be used. The program includes autoclave tests of both neat cement and mortar specimens, freezing and thawing of mortar specimens, and determinations of the air content and the absorption of mortars. Because this investigation cannot be completed for some time, it is recommended that the Tentative Specifications for Masonry Cement (C 91 – 44 T) be retained as tentative without change.

The Sponsoring Committee on Blended Cements (R. E. Roscoe, chairman) is engaged in the study of questions relating to pozzuolanic activity, and is also considering the use of the air permeability fineness test in place of the No. 325 sieve test that is now required by the Tentative Specifications for Portland – Blast Furnace Slag Cement (C 205 – 46 T). The subcommittee has proposed a revision affecting

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

Specifications C 205, as given later in this report.

The Working Committee on Methods of Chemical Analysis (W. C. Hanna, chairman) reports progress on a cooperative study of two methods of determining titanium oxide in portland cement. Eight laboratories are participating in the work. Certain changes in the Standard Methods of Chemical Analysis of Portland Cement (C 114 – 44) proposed by this subcommittee are given in the Appendix to this report. An informal arrangement has resulted in the formation of a joint committee consisting of three Government chemists and three members of Mr. Hanna's subcommittee. The purpose of this special subcommittee is to facilitate contact between the Federal Specifications Board and Committee C-1, and thereby promote uniformity in methods. The three Government chemists are B. L. Bean, In Charge Chemical Laboratory, Mineral Products Division, National Bureau of Standards; J. L. Gilliland, In Charge Chemical Laboratory, U. S. Bureau of Reclamation; H. A. Bright, Chief of Analytical Chemistry Section, National Bureau of Standards.

The Working Committee on Volume Change and Soundness (H. F. Gonnerman, chairman) is cooperating with the subcommittee appointed by Committee C-9 on Concrete and Concrete Aggregates to study chemical reactivity of aggregates of concrete. Mr. Gonnerman's subcommittee is devoting particular attention to the development of an acceptance test for use in determining the reactivity of aggregates. His subcommittee has also been engaged in assembling the data resulting from the examination of the 10-yr. old bars that had been used in the early autoclave studies.

The Working Committee on Time of Setting (E. E. Berger, chairman) is engaged in studies of certain proposed test methods. Seven laboratories are participating in the work.

The Working Committee on Bleeding, Plasticity, and Workability (H. L. Kennedy, chairman) presented an extensive report on studies of test methods that are within the subcommittee's field. The subcommittee has recently outlined an investigation of bleeding.

The Working Committee on Strength (G. L. Lindsay, chairman) has continued its investigation of problems relating to strength tests of portland cement and is studying the feasibility of using a fixed water-cement ratio in the mortar compression test. The subcommittee cooperated with other groups in studying tests for air-entrainment, and has proposed revisions for Method C 109 in the procedure for molding mortar cubes.

The Working Committee on Additions (H. J. Gilkey, chairman) has supervised the tests of a material that was submitted for determination of its acceptability as an addition to portland cement under the provisions contained in Specifications C 150. The tests, which are being made for Committee C-1 by the Cement Reference Laboratory, are almost completed.

The Working Committee on SO_3 Content (H. S. Meissner, chairman) is studying the question of how much the SO_3 limits can be raised in the portland-cement specifications and how performance tests might be made to determine the optimum SO_3 content for a given cement. The subcommittee has begun an extensive series of tests wherein eight cements, each ground with seven different SO_3 contents, are to be studied by a group of cooperating laboratories.

The new Working Committee on Methods of Test for Air-Entrainment (R. R. Litehiser, chairman) is investigating the details of apparatus and procedure involved in determining the air content of standard mortars. In this work the subcommittee is cooperating with other subcommittees that are interested in problems relating to air-entraining portland cement.

The Subcommittee on Cement Reference Laboratory (G. E. Warren, chairman) has continued its supervision of the work of the Cement Reference Laboratory at the National Bureau of Standards. That laboratory has continued its endeavors to promote uniformity and improvement in the testing of cement. The eighth tour of inspection of cement laboratories throughout the country was completed and the ninth tour was begun. Thus far, requests have been received for the inspection of 246 laboratories during the current tour. The laboratory completed the major part of a series of tests of a material proposed as an addition to cement, those tests being made for Committee C-1 under the supervision of the Working Committee on Additions. The laboratory cooperated with various subcommittees of Committee C-1, including the Working Committee on Coordination of Methods of Test (J. R. Dwyer, chairman) in matters relating to apparatus and methods.

I. REVISION OF TENTATIVES

Tentative Specifications for Air-Entraining Portland Cement (**C 175 - 46a T**).[1]—In keeping with revisions that are being recommended in the Standard Specification for Portland Cement (C 150 - 46), Committee C-1 recommends that the present requirement of "0.7 to 2.0" for the ratio of Al_2O_3 to Fe_2O_3 for type II A cement be deleted from Table I on Chemical Requirements of Specifications C 175. The committee also recommends that the limits on air content for types I A and II A cement in Table II be changed from "16 ± 4" to read "18 ± 3" per cent.

Tentative Specifications for Portland Blast-Furnace Slag Cement (**C 205 - 46 T**).[1]—The committee recommends that these specifications be revised to permit intergrinding TDA with the clinker and slag. The use of TDA is already permitted by the Standard Specifications for Portland Cement (C 150 - 46). The recommended revision is as follows:

Section 3 (*b*).—Add footnote 4, to read as follows: "The committee has declared as not harmful the inclusion of the material known commercially as TDA (composed of triethanolamine and highly purified soluble calcium salts of modified lignin sulfonic acids), manufactured by the Dewey & Almy Chemical Co., when added in an amount not to exceed 0.08 per cent by weight of the cement."

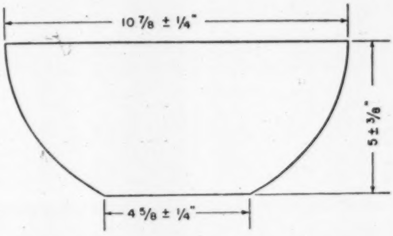

FIG. 1.—Mixing Bowl.

Tentative Method for Air Content of Portland Cement Mortar (**C 185 - 46 T**).[1]—Based on the results of subcommittee studies, Committee C-1 recommends the following revisions in this method in order to care for certain questions that had arisen concerning the Burmister flow trough:

Title.—Change to read: "Tentative Method of Test for Air Content of Air-Entraining Portland Cement Mortar."

Section 1.—Change to read: "This method of test is intended for determination of air content of air-entraining port-

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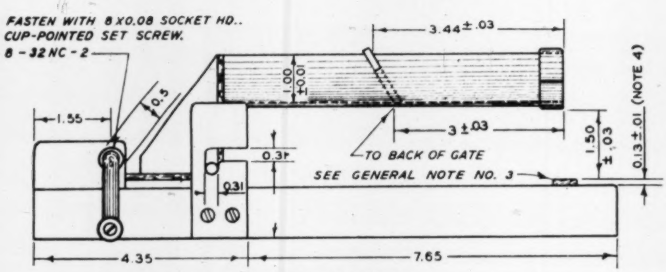
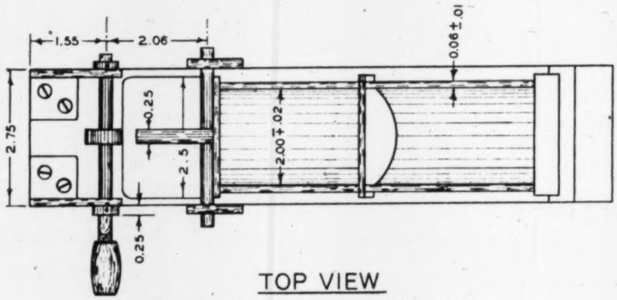
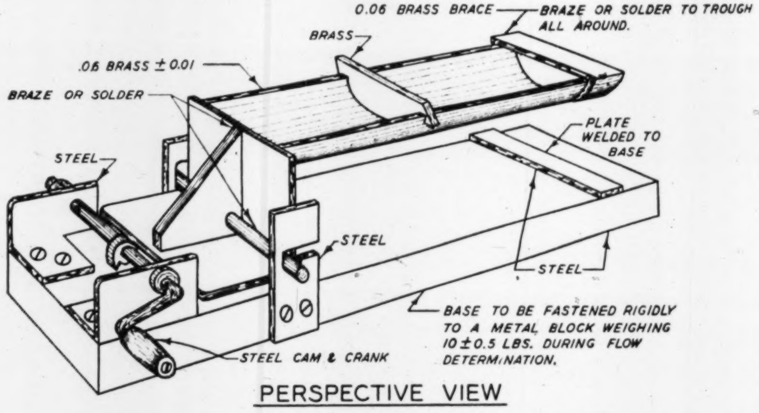
[1] 1946 Book of A.S.T.M. Standards, Part II.


FASTEN WITH 8 X 0.08 SOCKET HD.. CUP-POINTED SET SCREW.
8 - 32 NC - 2
3.44 ± .03
1.00 ± 0.01
0.5
1.55
3 ± .03
TO BACK OF GATE
0.31
0.31
SEE GENERAL NOTE NO. 3
1.50 ± .03
0.13 ± .01 (NOTE 4)
4.35
7.65

SIDE VIEW
(ASSEMBLY OF BASE AND TROUGH)


1.55
2.06
0.06 ± .01
2.75
0.25
2.5
2.00 ± .02
0.25

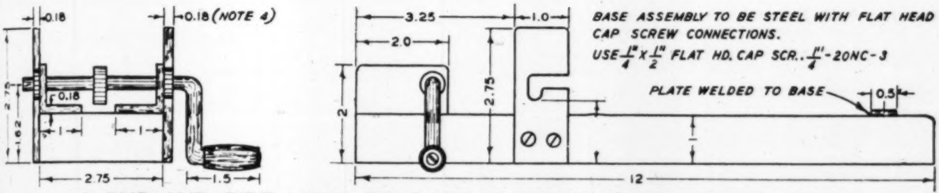
TOP VIEW
(ASSEMBLY OF BASE AND TROUGH)


0.06 BRASS BRACE
BRAZE OR SOLDER TO TROUGH ALL AROUND.
BRASS
.06 BRASS ± 0.01
BRAZE OR SOLDER
PLATE WELDED TO BASE
STEEL
STEEL
STEEL
BASE TO BE FASTENED RIGIDLY TO A METAL BLOCK WEIGHING 10 ± 0.5 LBS. DURING FLOW DETERMINATION.
STEEL CAM & CRANK

PERSPECTIVE VIEW

NOTE: THE TROUGH SHALL BE OF YELLOW BRASS WITH BRAZED OR SOLDERED CONNECTIONS. THE INNER SURFACE OF THE TROUGH SHALL HAVE AN UNPLATED SMOOTH, POLISHED FINISH.

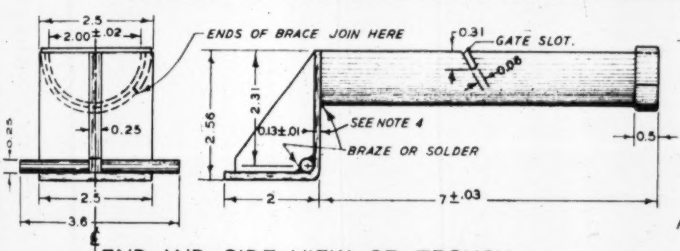
FIG. 2.—Burmister Mortar


0.18
0.18 (NOTE 4)
2.75
1.62
0.18
1
1
2.75
1.5
3.25
1.0
2.0
2
2.75
1
12
0.5

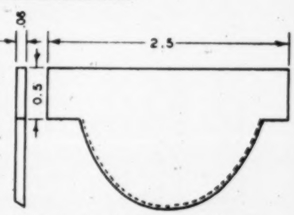
BASE ASSEMBLY TO BE STEEL WITH FLAT HEAD CAP SCREW CONNECTIONS.
USE $\frac{1}{4}$" x $\frac{1}{2}$" FLAT HD. CAP SCR. $\frac{1}{4}$"-20NC-3

PLATE WELDED TO BASE

END AND SIDE VIEW OF BASE SHOWING CAM AND CRANK

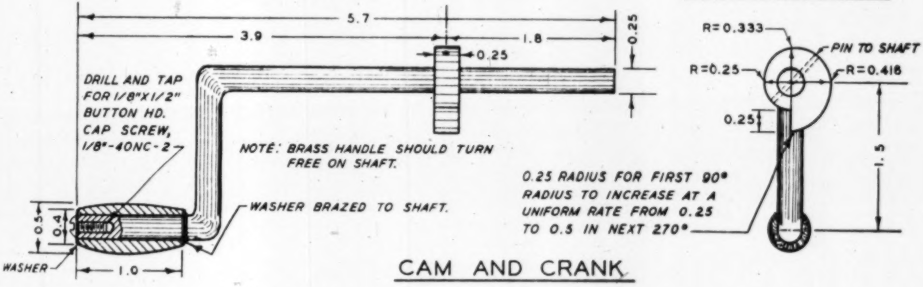

2.5
2.00±.02
0.25
0.25
2.5
3.6
℄
ENDS OF BRACE JOIN HERE
0.31
GATE SLOT.
0.08
2.31
2.56
0.13±.01
SEE NOTE 4
BRAZE OR SOLDER
0.5
2
7±.03

END AND SIDE VIEW OF TROUGH



NOTE: GATE TO BE FORMED TO FIT TIGHTLY INSIDE OF TROUGH AND TO FIT GATE SLOT SNUGLY.

VIEW OF GATE


5.7
3.9
1.8
0.25
0.25
0.5
0.4
WASHER
1.0
R=0.333
PIN TO SHAFT
R=0.25
R=0.418
0.25
1.5

DRILL AND TAP FOR 1/8"X1/2" BUTTON HD. CAP SCREW, 1/8"-40NC-2

NOTE: BRASS HANDLE SHOULD TURN FREE ON SHAFT.

WASHER BRAZED TO SHAFT.

0.25 RADIUS FOR FIRST 90° RADIUS TO INCREASE AT A UNIFORM RATE FROM 0.25 TO 0.5 IN NEXT 270°

CAM AND CRANK

General Notes

1. In the event that the balance point and/or weight of the trough is not within the prescribed tolerances, any weight added to or removed from the trough shall be added to or removed from the stiffener web or the heel of the trough in such a manner that the change in weight is symmetrical about the long axis of the trough.
2. The long axis of the trough shall be parallel with the base when the cam is at the point of release.
3. Plate to be located so that end of trough will strike center of plate.
4. $\frac{3}{16}$-in. stock is contemplated for the 0.18-in. trough support, and $\frac{1}{8}$-in. stock for the angle forming closed end of trough and the striking plate which is welded to the base.

land cement mortar under the conditions hereinafter specified."

Section 2(b).—Delete and make necessary redesignations of subsequent subsections.

Section 2(c).—Change to read: "A steel straightedge not less than $\frac{1}{16}$ in. nor more than $\frac{1}{8}$ in. in thickness."

Section 2(d).—Change to read: "A spatula with a metal blade 6 in. long and approximately $\frac{1}{2}$ in. wide with straight edges and a wooden handle."

Section 2(f).—Change to read: "A mixing bowl of 5 to 7 qt. capacity, made of stainless steel conforming in shape and dimensions to Fig. 1."

Section 2(h).—Change first sentence to read: "Glass graduates of 250-ml. capacities, with graduations in 2 ml., for measuring the mixing water, made to deliver the indicated volume at 20 C. (68 F.)."

Section 2(i).—Add the following as a second sentence: "The inner surface of the trough shall have an unplated, smooth polished finish." Change present second sentence to read: "The apparatus shall be constructed of materials and to the dimensions shown in Fig. 2." Delete from last sentence: "and free from set cement, dirt or grit." Add the following sentence at end of paragraph: "The base of the apparatus shall be fastened rigidly to a metal block weighing 10 ± 0.5 lb. during the flow determination." Add the following note at end of paragraph: "Note.—In the event that the balance point or weight of the trough is not within the prescribed tolerances, any weight added to or removed from the trough assembly shall be added to or removed from the stiffener web or the heel of the trough in such a manner that the change in weight shall be symmetrical about the long axis of the trough."

Section 5(a).—Change: "4-ml." to "3-ml."

Section 5(b).—Change second sentence to read: "No stirring action shall be employed except occasional sweeps of the gloved hand to remove mortar from sides of the bowl and bringing it into a pile for the squeezing and kneading operation."

Section 5(c).—Delete from ninth sentence (line 9, column 1, 1946 Book of A.S.T.M. Standards, Part II, p. 1280) the words: "of the spatula."

Section 5(d).—Delete the third, fourth and fifth sentences. Replace by the following: "The mortar shall be placed in the 500-ml. measure in three equal layers, spading each layer thoroughly with the spatula around the inner surface of the measure. After the measure has been filled and spaded in the above prescribed manner, the sides of the measure shall be tapped lightly five times with the wooden handle of the spatula in order to preclude entrapment of extraneous air. Care shall be taken that no space is left between the mortar and the inner surface of the measure as a result of the spading operation."

Section 6.—Change to read: "The air content of the mortar shall be calculated from the following formula, which is based on use of the measure specified in Section 2(*a*) and on the batch proportions given in Section 5(*a*)."

Figure 1.—Replace by the accompanying new Fig. 1 and add note as follows: "Bowls of this type should be available at restaurant supply houses."

Figure 2.—Present Fig. 1 revised. The revisions proposed in the accompanying Fig. 2 are intended to secure appropriate tolerances in dimensions, clarify details that have been the subjects of recent questions, and effect certain desirable improvements.

II. REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends, for immediate adoption, revisions in the following two standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Standard Specifications for Portland Cement (**C 150 – 46**):[1]

Table I.—Delete the present requirement of "0.7 to 2.0" for the ratio of Al_2O_3 to Fe_2O_3.

In the requirements for type V cement delete the existing limit of "24.0" per cent for silicon dioxide and in its stead add a limit of "50" per cent to the tricalcium silicate content as calculated by the usual formula; change the limit for ferric oxide in type V cement from "4.0" to read "5.0" per cent. These changes will make the manufacture of type V cement a more practical and economical procedure, thereby making that cement more generally available than it has been heretofore. Moreover, the revised limits for type V cement would be in closer accord with the Federal Specification SS-C-192.

Standard Methods of Chemical Analysis of Portland Cement (**C 114 – 46**):[1]

The committee recommends for immediate adoption the revisions that were proposed by the Working Committee on Methods of Chemical Analysis as given in the Appendix to this report. Some of these changes have been incorporated in the methods in the Federal Specification for Portland Cements (SS-C-158b). Attention is invited to the precaution conveyed by the proposed addition to Note 1 of Section 46.

III. ADOPTION OF TENTATIVE AS STANDARD

Tentative Method of Test for Heat of Hydration of Portland Cement (**C 186 – 44 T**).[1]—This method having now stood as tentative without change for three years, Committee C-1 recommends that it be approved for reference to letter ballot of the Society for adoption as standard without revision.

IV. ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends the adoption as standard of the tentative revision of the Standard Method of Test for Compressive Strength of Hydraulic Cement Mortars (C 109 – 44.)[2]

The recommendations appearing in this report have been submitted to letter ballot[3] of the committee, the results of which will be reported at the Annual Meeting.

This report has been submitted to letter ballot of the committee, which consists of 85 voting members; 64 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. H. JACKSON,
Chairman.

G. E. WARREN,
Secretary.

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[2] 1946 Book of A.S.T.M. Standards, Part II, p. 1701.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

APPENDIX

PROPOSED REVISIONS IN THE STANDARD METHODS OF CHEMICAL ANALYSIS OF PORTLAND CEMENT (C 114 - 46)[1]

Footnote 6.—Add the following to the last sentence: "or AnalaR Standards for Laboratory Chemicals, The British Drug House, Ltd. and Hopkin and Williams, Ltd., London (1944)."

Section 7.—Add the following new Paragraphs (*c*) and (*d*):

(*c*) *Referee Methods.*—The methods covered in sections 8 to 32 shall be known as referee methods. The cement shall not be rejected for failure to conform to a chemical requirement unless the chemical determination is made by a procedure classified as a referee method (Note 2) and as prescribed in this paragraph. The determination shall be made in duplicate and the analyses shall be made on different days. If the two results do not agree within the permissible variation given in Table I, the determination shall be repeated until two or three results agree within the permissible variation. When two or three results do agree within the permissible variation, their average shall be accepted as the correct value. When either an average of two results or an average of three results can be calculated, the calculation shall be based on the three results. For the purpose of comparing analyses and calculating the average of acceptable results, the percentages shall be calculated to the nearest 0.01 (or 0.001 in the case of chloroform-soluble organic substances), although some of the average values are reported to 0.1 as indicated in the methods. When a blank determination is specified, one shall be made with each individual analysis or with each group of two or more samples analyzed on the same day for a given component.

TABLE I.—MAXIMUM PERMISSIBLE VARIATIONS IN RESULTS.

Component	Between Two Results	Between the Extreme Values in Three Results
Silicon dioxide, SiO_2	0.16	0.24
Aluminum oxide, Al_2O_3	0.20	0.30
Ferric oxide, Fe_2O_3	0.10	0.15
Calcium oxide, CaO	0.20	0.30
Magnesium oxide, MgO	0.16	0.24
Sulfur trioxide, SO_3	0.10	0.15
Sulfide sulfur, S	....	....
Loss on ignition	0.10	0.15
Sodium oxide, Na_2O	0.03	0.05
Potassium oxide, K_2O	0.03	0.05
Water-soluble alkali	0.05	0.08
Phosphorus pentoxide, P_2O_5	0.03	0.05
Manganic oxide, Mn_2O_3	0.03	0.05
Insoluble residue	0.10	0.15
Chloroform-soluble organic substances	0.004	0.006
Free calcium oxide	0.20	0.30

Note 2.—Referee methods are so called because they are required for referee analysis, but it is not intended that their use be confined to referee analysis. A referee method may be used in preference to an optional method when a question of rejection is not involved. A referee method must be used where an optional method is not provided.

(*d*) *Optional Methods.*—The methods covered in Sections 33 to 52 shall be known as optional methods and may be used when a chemical determination conforms to a specification requirement. They are provided for those who wish to use procedures shorter or more convenient than the referee methods for the routine determination of certain components. Duplicate analysis and blank determinations are not required when a chemical determination conforms to a specification requirement. Under the same conditions, in the place of the omission of the blank determinations, a blank determination may be used to correct the results of analyses not made concurrently with the blank determination.

Sections 8, 9, and 12.—Change all references to "aluminum oxide and ferric oxide" or "$Al_2O_3+Fe_2O_3$" to read "ammonium hydroxide group." Correct the methods throughout to conform to this change.

Section 8 (*c*).—Omit the word

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[1] For explanation of this revision, see p. 231.

"weighed" in the first sentence, add "(Note 2)" at the end of the sentence and add the following new note:

NOTE 2.—The empty crucible may be weighed if one wishes to know, for his own information, the magnitude of impurities in the residue of SiO_2.

Section 8 (*d*).—Change the last sentence to read:

To this amount of SiO_2, add the amount of SiO_2 recovered from the residue of ammonium hydroxide group as directed in Section 9(*d*) and (*e*). Add 0.5 g. of $Na_2S_2O_7$ or $K_2S_2O_7$ to the crucible and heat below red heat until the small residue of impurities is dissolved in the melt. Cool, dissolve the fused mass in water, and add it to the filtrate and washings reserved for the determination of the ammonium hydroxide group.

Section 9 (*a*).—Change Paragraph (*a*) to read as follows:

(*a*) To the filtrate reserved in accordance with Section 8(*b*) (Note 1), which should have a volume of about 200 ml., add HCl if necessary to insure a total of 10 to 15 ml. of the acid. Add a few drops of methyl red indicator (2 g. per liter of ethanol), and heat to boiling. Then treat with NH_4OH (1:1) (Note 2), drop by drop, until the color of the solution becomes distinctly yellow and add one drop in excess (Note 3). Bring to boiling the solution containing the precipitate and boil for 50 to 60 sec. Allow the precipitate to settle (in not more than 5 min.) and filter. Wash, from two times for a small precipitate, to four times for a large one, with hot NH_4Cl (20 g. per l.).

Add the following new note:

NOTE 3.—It usually takes one drop of NH_4OH (1:1) to change the color of the solution from red to orange and another drop to change the color from orange to yellow. If desired, the addition of the indicator may be delayed until Fe $(OH)_3$ is precipitated without $Al(OH)_3$ being completely precipitated. In such a case, the color changes may be better observed. However, if the content of Fe_2O_3 is unusually great, it may be necessary to let the precipitate settle a little, a few times, before the proper end point is attained. If the color fades during the precipitation, add more of the indicator. Observation of the color where a drop of the indicator strikes the solution may be an aid in the control of the acidity. The boiling should not be prolonged as the color may reverse and the precipitate may be difficult to retain on the filter. The solution should be distinctly yellow when it is ready to filter. If it is not, restore the yellow color with more NH_4OH (1:1) or repeat the precipitation.

Section 9 (*d*).—Replace the present Paragraph (*d*) by the following new Paragraphs (*d*) and (*e*):

(*d*) Add 3 g. of $Na_2S_2O_7$ or $K_2S_2O_7$ to the crucible (Note 4) and heat below red heat until the residue is dissolved in the melt (Note 5). Cool, dissolve the fused mass in water containing 2.5 ml. of H_2SO_4, and evaporate the solution. Raise the temperature until copious fumes are evolved, but avoid a large loss of H_2SO_4 which will cause the mass to become hard instead of pasty on cooling. Dissolve the mass in water, digest for 15 to 30 min. short of boiling, filter, and wash with hot water.

NOTE 4.—The procedure described in paragraphs (*d*) and (*e*) is required only when a chemical determination fails to meet a specification requirement.

NOTE 5.—Start the heating with caution because pyrosulfates (also known as fused bisulfates) as received often foam and spatter in the beginning, due to an excess of H_2SO_4. Avoid an unnecessarily high temperature and unnecessarily prolonged heating as fused pyrosulfates may attack platinum. A supply of nonspattering pyrosulfate may be prepared by heating some pyrosulfate in a platinum vessel below red heat until the foaming and spattering cease, cooling, and crushing the fused mass.

(*e*) Transfer the paper (Note 4) containing the residue to a platinum crucible. Dry and ignite the paper, first at a low heat until the carbon of the paper is completely consumed without flaming, and finally at 1100 to 1200 C. until the weight remains constant. Treat the SiO_2 thus obtained in the crucible with a drop of water, about 5 ml. of HF, and a drop of H_2SO_4, and evaporate cautiously to dryness. Finally, heat the crucible at 1050 to 1100 C. for 1 or 2 min., cool, and weigh. The difference between this weight and the weight previously obtained represents the amount of residual SiO_2. Subtract this amount from the amount of ammonium hydroxide group obtained according to Paragraph (*c*) and add the same amount to the amount of SiO_2 obtained according to Section 8 (*d*).

Section 9 (*f*).—Change "to the nearest 0.1" to read "to the nearest 0.01."

Section 10 (a).—Change the first sentence of this paragraph to read "Dissolve 5 g. of $SnCl_2 \cdot 2H_2O$ in 10 ml. of HCl and dilute to 100 ml."

Section 10 (b) and (c).—Delete Paragraph (*b*) and change the present Paragraph (*c*) to read as follows: "(*b*) *Barium Diphenylamine Sulfonate Indicator.*—Dissolve 0.3 g. of barium diphenylamine sulfonate in 100 ml. of water."

Section 10 (d).—Change the second sentence of this paragraph to read as follows:

> Standardize against standard Sibley iron ore issued by the National Bureau of Standards (standard sample No. 27 or its replacements) in the same manner (Note) as directed for the determination of Fe_2O_3 in cement (Section 11), using a weight of iron ore that will yield a titration within 3 ml. of that required by the cement sample in question.

In the third sentence, change "iron wire" to "iron ore." Add the following new note:

> NOTE.—The iron ore may require long digestion in hot HCl for complete dissolution. Stannous chloride may be used as an aid in the dissolution, as follows: Treat the sample with 15 ml. of HCl and heat short of boiling for about 30 min. Add some of the $SnCl_2$ solution, using less than the amount that is expected to reduce all the iron. Continue the digestion until the iron is dissolved out, as evidenced by the absence of dark residue. The $SnCl_2$ may be added in small quantities during the digestion. If an excess is present at the end of the digestion, destroy it with bromine water. Dilute the solution to about 50 ml., heat the solution to boiling, and proceed as directed in Section 11, beginning with decolorization with $SnCl_2$.

Section 11 (a).—In the first sentence change "15 ml. of HCl" to read "10 ml. of HCl."

Change the sixth sentence to read: "Stir the solution vigorously for 1 min. and add 10 ml. of H_3PO_4 (1:1) and 2 drops of barium diphenylaminesulfonate indicator. Add sufficient water so that the volume after titration will be between 75 and 100 ml."

In the last sentence, change "deep blue" to read "purple."

Section 11 (b).—Change "to the nearest 0.1" to read "to the nearest 0.01 (to be reported to the nearest 0.1)."

Section 12.—Change to read as follows:

> 12. *Calculation.*—Calculate the percentage of Al_2O_3 by deducting the percentages of Fe_2O_3 and P_2O_5, determined according to Sections 11 and 21 to 22, respectively, and expressed to the nearest 0.01, from the percentage of ammonium hydroxide group, determined according to Section 9 and expressed to the nearest 0.01. Report to the nearest 0.1.
>
> NOTE.—The determination of P_2O_5 and its deduction from the determination of ammonium hydroxide group are required only when a sample fails to meet a specification requirement. Any titanium that may be present will be precipitated with $Al(OH)_3$ and $Fe(OH)_3$ in the procedure described in Section 9 and is counted as Al_2O_3. Aluminum oxide should not be corrected for TiO_2 unless such a correction is expressly specified or the determination of the component is required.

Section 13 (a).—Change the sixth sentence from its present form "Discard the precipitate" to read "Discard any manganese dioxide that may have been precipitated."

Section 15 (b).—Insert "(Note)" after "1100 to 1200 C." and add the following new note:

> NOTE.—If the crucible is glazed, the temperature should not exceed 1125 C. as the glaze may partially fuse at about 1200 C.

Section 16 (a).—Insert "(Note 1)" at the end of the second sentence.

Change the third sentence to read as follows: "Dilute the solution to 50 ml. and digest for 15 min. short of boiling."

Change the seventh sentence to read as follows: "Digest the solution for 12 to 24 hr. short of boiling (Note 2). Take care to keep the volume of solution between 225 and 260 ml. and add water for this purpose if necessary."

Add the following new notes:

NOTE 1.—A brown residue due to compounds of manganese may be disregarded. See Note Section 28.

NOTE 2.—If a rapid determination is desired, the time of digestion may be reduced to as little as 3 hr. The result may be slightly low. If the cement is rejected for failure to meet the specification requirement, the time of digestion shall be 12 to 24 hr.

In the eighth sentence, add "or porcelain" after "platinum."

Change the last sentence to read as follows: "Then ignite at 800 to 900 C., cool in a desiccator, and weigh the $BaSO_4$."

Section 21 (*a*).—Change to read as follows: "(*a*) *Chloroplatinic Acid Solution.*—Dissolve 5 g. of $H_2PtCl_6 \cdot 6H_2O$ in 95 ml. of water and 5 ml. of HCl."

Section 22 (*a*).—In the last sentence, change "ignite the crucible at 900 to 1000 C." to read "ignite the crucible so that the temperature of the contents will be maintained at 900 to 1000 C."

Section 30 (*a*).—Change Paragraph (*a*) to read as follows:

(*a*) Place 40 g. of cement in a 1-liter Squibb separatory funnel (Note 1) and mix it with 520 ml. of water added in two approximately equal portions. Shake vigorously immediately after the addition of the first portion to effect complete dispersion. Then add the second portion and shake again. At once add rapidly 185 ml. of HCl in which 10 g. of $SnCl_2$ (Note 2) have been dissolved, rapidly insert the stopper in the funnel, invert, and shake with a swirling motion for a few seconds to loosen and disperse all the cement, taking care to avoid the development of great internal pressure due to unnecessarily violent shaking. Release internal pressure immediately by opening and closing the stopcock. Repeat the shaking and release of pressure until the decomposition of the cement is complete. If necessary, break up persistent lumps with a long glass rod. Cool to room temperature rapidly by allowing tap water to run on the flask. Add 75 ml. of chloroform to the solution, stopper the funnel, shake it vigorously for 5 min., and allow the water and chloroform to stand 15 min. to separate.

Add the following new note:

NOTE 2.—The purpose of the $SnCl_2$ is to prevent the oxidation of sulfide sulfur to elemental sulfur, which is soluble in chloroform, but if Vinsol resin is present, its methoxyl content may be reduced by $SnCl_2$. When the extraction with chloroform is made for the purpose of making a determination of Vinsol resin based on its methoxyl content according to Sections 2 to 4 of the Tentative Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114 – 46 T), $SnCl_2$ should be omitted.

Section 30 (*d*).—At the end of Note 5, add the following as a new paragraph:

When Vinsol resin is known to be the only substance present, the residue may be heated at 100 to 105 C., instead of 57 to 63 C., in order to expel all possible traces of chloroform.

Section 33 (*b*).—Add "(Note 3)" at the end of the paragraph, and add the following new note:

NOTE 3.—If there is any doubt about the percentage of SiO_2 meeting a specification requirement, the amount of impurities in the residue may be ascertained by treating the SiO_2 with HF and H_2SO_4 as directed in Section 8(*d*). However, the cement shall not be rejected for a low SiO_2 content unless the determination is made according to Section 8.

Section 35.—Add "(Note 1)" after "filtrates" in the first sentence and add the following new note:

NOTE 1.—If there is a possibility of there being enough manganese to cause the percentage of MgO, as determined by Section 36, to exceed a specified limit, manganese may be eliminated from the filtrate before the directions of Section 35 are followed. Although the elimination may be made as directed in Section 13(*a*), the cement shall not be rejected for exceeding a limit specified for MgO unless the determination of MgO is made according to Section 15.

Section 39 (*b*).—In the second sentence, change "either" to read "directly against $K_2Cr_2O_7$, or." In the third sentence, change "Nos. 40 and 83" to "Nos. 136, 40, and 183."

Section 40 (*a*).—After "cement" in the first sentence add "(Note 1)." Add the following as a new note:

NOTE 1.—If SiO_2, ammonium hydroxide group, and CaO are separated and determined according to Section 8 or 33, Section 9, and Section 13 or 34, respectively, the remaining filtrate may be used for the determination of MgO by Section 40(*c*). However, the cement shall not be rejected for failure to meet a requirement for MgO unless this component is determined according to Section 15.

Section 40 (*b*).—Change the eighth sentence to read as follows: "Combine this filtrate and washings with those from the first precipitation and add 1 ml. of NH_4OH."

Section 46.—At the end of Note 1, add the following sentence: "If the cement is known or suspected to contain organic matter, destroy the organic matter by spreading the sample in the platinum dish in a thin layer and igniting the cement at 600 to 700 C. for 10 to 15 min. before the treatment with water and acid."

REPORT OF COMMITTEE C-7*

ON

LIME

Committee C-7 on Lime held two meetings during the past year. The first was held on June 25, 1946, in Buffalo, N. Y., during the Annual Meeting of the Society. At this meeting a special committee was appointed to redraft the By-laws and to report back to the committee at its next meeting.

A second meeting of the committee was held on February 25, 1947, in Philadelphia, Pa., at which the desirability of preparing master specifications for the chemical lime industries was discussed. The committee voted to review and revise existing specifications rather than prepare a master specification and Subcommittee III was requested to consider the formulation of tentative specifications for chemical lime used in other industries but not now covered.

The report of the Subcommittee on By-laws was discussed and it was voted to send the revised By-laws, as amended in part at this meeting, to letter ballot.

The committee now consists of 45 members, of whom 21 are classified as producers, 4 as consumers, and 20 as general interest members.

ADOPTION OF TENTATIVE REVISIONS AS STANDARDS

Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (**C 25 - 44**).[1]—A tentative revision[2] of this method, which consists of adding a new section on unhydrated oxides calculated on the as-received basis, has been published since June, 1945. The committee recommends that this tentative revision be approved for reference to letter ballot of the Society for adoption as standard.

Standard Definitions of Terms Relating to Lime (**C 51 - 44**).[1]—The committee recommends that the tentative revision of this standard, published since June, 1944, in the form of Tentative Definitions of Terms Relating to Lime (C 51 - 44 T),[3] be approved for reference to letter ballot of the Society for adoption as standard and added to the present standard definitions.

The recommendations in this report have been submitted to letter ballot[4] of the committee, the results of which will be reported at the Annual Meeting.

This report has been submitted to letter ballot of the committee, which consists of 45 members; 33 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

WALTER C. VOSS,
Chairman.

ROBERT S. BOYNTON,
Acting Secretary.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] 1946 Book of A.S.T.M. Standards, Part II.

[2] *Ibid.*, p. 1701.

[3] *Ibid.*, p. 1319.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

REPORT OF COMMITTEE C-8* ON REFRACTORIES

Committee C-8 on Refractories held two meetings during the year, at Battelle Memorial Institute, Columbus, Ohio, on November 15, 1946, and at the Department of Commerce Building, Washington, D. C., on March 26, 1947.

New members elected during the year were: A. W. Allen, University of Illinois; H. A. Heiligman, E. J. Lavino and Co.; and E. B. Snyder, Wheeling Steel Corporation. Because R. B. Sosman changed his affiliation and is now on the staff of Rutgers University, a reaffirmation of his membership was necessary. A. S. Klopf of the Western Foundry Co. was appointed by the American Foundrymen's Association as its representative on Committee C-8; C. B. Bradley of Johns-Manville resigned because of pressing duties and requested the election of W. L. Stafford to replace him on the committee, which was approved.

I. New Tentative

The committee recommends that the new proposed Tentative Specifications for Castable Refractories for Boiler Furnaces and Incinerators[1] be accepted for publication as tentative. Castable refractory materials find extensive use in the construction and maintenance of boiler furnaces and incinerators. This class of refractory material has been in use for many years, and although the industry, state, and Federal governments have specifications for the material, Committee C-8 has not until recently had sufficient technical information to prepare satisfactory specifications. These specifications are the result of an investigation of the properties of 12 brands of widely used commercial castable refractories and a survey of the requirements of castable refractories in the two types of furnaces involved. It incorporates a class for service at 2400 F. and one for 2700 F. Furthermore, the material may be supplied in the usual grind or particle size but, in addition, a finer grind material may be stipulated when the thickness of the wall to be cast is of the order of 2 in. or less. The test methods used for evaluation are A.S.T.M. standard methods modified where necessary for use with castable refractories.

II. Tentative Revisions of Standards

Standard Method of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes (**C 133 – 39**).[2]—The committee recommends the following sentence for publication as a tentative revision:

Sections 5 (*d*) *and 9* (*c*).—Add the following sentence to the end of the paragraph: "A tolerance of plus or minus 10 per cent shall be permitted in the rate of application of load with either the mechanical or hydraulic testing machine."

This is an additional refinement to the procedure which should help to eliminate a possible source of error. Furthermore, this change brings Method C 133 in line with a similar requirement incorporated in the Standard Methods of Test for Crushing Strength and Modulus of Rupture of Insulating Fire Brick at Room Temperature (C 93 - 46), which pertains to the same test as applied to insulating fire brick.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] These specifications were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part II.

[2] 1946 Book of A.S.T.M. Standards, Part II.

Standard Definitions of Terms Relating to Refractories (**C 71 - 46**).[2]—Since the need for definitions covering additional terms relating to refractories has been expressed, the committee recommends the following definitions for publication as tentative revisions:

Dead-Burned Magnesite.—The granular product obtained by burning (firing) magnesite or other substances convertible to magnesia, upon heating above 1450 C. long enough to form dense, weather-stable granules suitable for use as a refractory or in refractory products.

Refractory Chrome Ore.—A refractory ore consisting essentially of chrome-bearing spinel with only minor amounts of accessory minerals and with physical properties that are suitable for making refractory products.

Chrome Brick.—A refractory brick manufactured substantially or entirely of chrome ore.

Magnesite Brick.—A refractory brick manufactured substantially or entirely of dead-burned magnesite.

The present definition for "burning (firing) of refractories" has been pointed out as being somewhat misleading inasmuch as it might include fused cast refractories. To overcome this ambiguity, a tentative revision is recommended, involving the insertion of "in a kiln" after the words "heat treatment" in the definition.

Standard Specifications for Refractories for Heavy Duty Stationary Boiler Service (**C 64 - 41**),[2] *Refractories for Incinerators* (**C 106 - 41**),[2] and *Refractories for Moderate Duty Stationary Boiler Service* (**C 153 - 41**).[2]—The modulus of rupture requirement for the low-heat duty, type G, brick as incorporated in Section 6 (*g*) of Specifications C 64, Section 5 (*c*) of Specifications C 106, and Section 5 (*b*) of Specifications C 153 stipulates a strength of not less than 500 psi. Because of the service requirements in the specifications for this class of refractory, together with the properties of the present-day product, the committee recommends for publication as a tentative revision of each of the above mentioned standard specifications that the strength limit be increased from "500" to read "800" psi.

Standard Method of Test for Fireclay Refractories Under Load at High Temperatures (**C 16 - 41**).[2]—This method has been critically reviewed and, as a result, several changes of a minor nature pertaining to the procedure are recommended, as well as the inclusion of heating schedules for the testing of silica and basic and neutral refractories. A number of years ago this procedure incorporated a schedule for silica brick, but at that time the temperature of the test was not sufficiently high to provide a satisfactory evaluation of silica refractories. In recent years, use has been made of the load test for obtaining the refractoriness of silica, neutral and basic refractories, by obtaining the temperature of failure by shearing of the brick under a load of 25 psi. This is in contrast with the present procedure for testing fire-clay refractories under load where the heat is raised to a specified temperature and maintained for 1½ hr. to obtain the percentage of deformation. The time-temperature schedule for use in obtaining the shearing temperature of the two types of refractories mentioned has been carefully worked out so as to comply with the properties of each and, at the same time, enabling the test to be carried out in an 8-hour day. The committee recommends the following revised paragraphs and new subject matter for publication as tentative revisions of this method:

Section 2 (*b*).—Change the last sentence to read as follows: "If the optical pyrometer is used, observations shall be made by sighting on to the face of the specimens and in the same relative positions as those specified for the thermocouples."

At present the optical pyrometer sighting is to be directed into a closed and refractory tube projecting into the furnace, but this practice has not proved practical because of inability to obtain tubes of sufficient refractoriness for the purpose and, consequently, that detail of the method has not been followed to any extent.

Section 5 (a).—Add the following to the end of this paragraph: "When testing specimens that are likely to fail by shear provision should be made so that the loading mechanism cannot drop more than ½ in. when failure occurs."

Section 5 (d).—Delete this paragraph and substitute the following:

> (*d*) *Completion of Test.*—At the expiration of the time of heating, the supply of heat shall be stopped and, in the case where specimens have not failed, the furnace shall be allowed to cool by radiation to 1830 F. (1000 C.) or lower before the load is removed and the specimens examined. When failure by shearing has not occurred, the test specimens shall be remeasured, after cooling to room temperature, for length in accordance with Section 3(*c*) and the average deformation, based on the original length, shall be reported to the nearest 0.5 per cent. When brick fail by shear, the temperature of shear shall be recorded and reported.
>
> NOTE.—It is recommended that a photograph be made of the specimens before and after testing as yielding useful information.

The two changes in Section 5 are to provide instructions necessitated by the incorporation of the new heating schedules for the shear temperature as the end point of the test.

Table I.—Revise the present Table I, providing three time-temperature schedules for fireclay refractories, to incorporate the new schedule for silica and basic and neutral brick as schedules 4 and 5. These are to be as follows:

Elapsed Time from Start of Heating		Temperatures for Various Heating Schedules			
Hours	Minutes	Schedule No. 4 Silica Brick		Schedule No. 5 Basic and Neutral Brick[a]	
		deg. Fahr.	deg. Cent.	deg. Fahr.	deg. Cent.
1	0	245	119	930	500
	15	310	154	1150	620
	30	380	193	1330	720
	45	450	232	1500	815
2	0	535	280	1650	900
	15	630	335	1795	980
	30	775	413	1915	1045
	45	1025	552	2010	1100
3	0	1275	690	2100	1150
	15	1525	830	2185	1195
	30	1750	955	2255	1235
	45	1990	1090	2320	1270
4	0	2200	1205	2370	1300
	15	2400	1315	2425	1330
	30	2550	1400	2460	1350
	45	2650	1455	and to failure at 100 F. (56 C.) per hr.	
5	0	2700	1480		
		and to failure at 100 F. (56 C.) per hr.			

[a] This schedule is for testing neutral and basic brick which are expected to show failure below 2900 F. (1595 C.).

In addition to schedule No. 5, there is need for one which will reach at least 3200 F. for use in evaluating other types of basic brick and super duty special refractories. A proposal for this will be submitted at a later time after full consideration can be given to the rate of heating requirements, particularly with respect to the completion of the test within a working day. Mention should be made that appropriate editorial changes are also being made in the procedure which are in keeping with the shear method of testing and details relating to current practice.

III. ADOPTION OF TENTATIVES AS STANDARD

Because the committee has not received noteworthy criticism or constructive suggestions on the following tentatives, Committee C-8 recommends that they be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Method of Test for:

Bonding Strength of Air-Setting Refractory Mortar (Wet Type) (C 198 - 45 T),
Refractoriness of Air-Setting Refractory Mortar (Wet Type) (C 199 - 45 T),
Thermal Conductivity of Insulating Fire Brick (C 182 - 45 T),
Thermal Conductivity of Refractories (C 201 - 45 T), and
Thermal Conductivity of Fireclay Refractories (C 202 - 45 T).

Tentative Specifications for:

Air-Setting Refractory Mortar (Wet Type), for Boiler and Incinerator Service (C 178 - 45 T), and
Classification of Insulating Fire Brick (C 155 - 45 T).

IV. ADOPTION OF TENTATIVE REVISIONS AS STANDARD

Standard Definitions of Terms Relating to Refractories (**C 71 - 46**).—This standard contains a definition for fire clay which has been subject to considerable criticism. A new definition was submitted as a tentative revision[2] in June, 1946. Since that time, however, the following revised definition has been prepared and approved, and the committee accordingly recommends that it be adopted as standard:

> *Fire Clay.*—An earthy or stony mineral aggregate which has as the essential constituent hydrous silicates of aluminum with or without free silica, plastic when sufficiently pulverized and wetted, rigid when subsequently dried, and of suitable refractoriness for use in commercial refractory products.

Such action is requested because of the belief that the unusual amount of attention devoted to the preparation of the definition has resulted in a satisfactory draft, and it should be adopted as standard in place of the tentative revision submitted in June, 1946.

The present definition for air-setting refractory mortar has been the subject of criticism and the committee now has an improved definition as a tentative revision to replace the present one. Because of not receiving noteworthy criticism, it is recommended that this tentative be adopted as standard.

Standard Methods of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (**C 38 - 45**), *Panel Test for Resistance to Thermal and Structural Spalling* (**C 107 - 45**), *Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick* (**C 122 - 45**), and *Panel Test for Resistance to Thermal and Structural Spalling of Fire Clay Plastic Refractories* (**C 180 - 45**).—Each of these four methods pertaining to the panel spalling test on various grades and types of refractories has a paragraph relating to treatment of the test brick after the spalling operation for the purpose of obtaining the actual spalling loss. This paragraph does not explain the operations involved in as much detail as should be the case. A revised and suitable paragraph was submitted as a tentative revision in June, 1946. The committee now recommends that these tentative revisions be adopted as standard.

Standard Specifications for Refractories for Malleable Iron Furnaces with Removable Bungs and for Annealing Ovens (**C 63 - 41**), *Refractories for Heavy Duty Stationary Boiler Service* (**C 64 - 41**) *Refractories for Incinerators* (**C 106 - 41**), and *Refractories for Moderate Duty Stationary Boiler Service* (**C 153 - 41**).—The committee also recommends that the tentative revisions for these standard methods relating to a paragraph and table which provide information for the consumer for the selection of the required samples necessary for conducting the tests involved in each specification, submitted in June, 1946, be adopted as standard.

Standard Method of Test for True Specific Gravity of Burned Refractory Materials (**C 135 - 40**).—The tentative revision of this method involves an improved presentation which provides for the testing of samples in the pulverized condition, demands closer limits for the temperature of the water bath, and includes a boiling procedure as a permissible alternate. It is recommended that this tentative revision be adopted as standard.

Specifications for Fireclay Plastic Refractories for Boiler and Incinerator Services (**C 176 - 44**).—The tentative revision of these specifications relates to an additional paragraph which specifies the size and selection of the sample for use in conducting the tests involved in the specifications. The committee recommends that this tentative revision be adopted as standard.

Standard Method of Test for Workability Index of Fireclay Plastic Refractories (**C 181 - 45**).—The tentative revision of this test method pertains to the mounting of the rammer used in the test on a specified base of substantial weight for the purpose of eliminating variables. The committee recommends that this tentative revision be adopted as standard.

Standard Specifications for Ground Fire Clay as a Mortar for Laying-Up Fireclay Brick (**C 105 - 41**).—In 1944, a tentative revision of these specifications was approved but inadvertently has not as yet been recommended for adoption as standard. This pertains to the inclusion of a retest paragraph of suitable wording and which, incidentally, is incorporated in all the other specifications of this committee. The committee recommends that this tentative revision be adopted as standard.

V. STANDARD CONTINUED WITHOUT REVISION

Standard Classification of Fireclay Refractories (**C 27 - 41**).—The committee has not received noteworthy criticisms which could be used in improving the substance or principle involved in this classification of fireclay refractories. A unanimous reaffirmation vote recommends that the classification be continued as standard.

The recommendations appearing in this report were submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (W. S. Debenham, chairman).—The chairman of the subcommittee has prepared a revision of the open-hearth survey which will be incorporated in the forthcoming edition of the Manual of A.S.T.M. Standards on Refractory Materials of the Society. Efforts are being made by the subcommittee to obtain revisions of the present surveys relating to coke ovens and the lead industry. Desirable subjects for new surveys pertain to water-gas machines and steel pouring-pit refractories, and an endeavor is being made to obtain authors for these subjects.

Subcommittee II on Research (R. B. Sosman, chairman).—For the purpose of encouraging study on selected subjects relating to refractories, the subcommittee has submitted a brief description of six problems to the *Bulletin* of the American Ceramic Society for publication and copies will be sent later to certain individuals to further the purpose. Active work is being carried out in at least four laboratories on the study of creep of refractories, and the report of one such investigation has been published by the National Bureau of Standards. Studies have shown that the spectrographic method of analysis is applicable for the determination of alumina in ganister and silica refractories.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee III on Tests (S. M. Phelps, chairman).—This committee is comprised of seven sections which look after test procedures falling in specific categories. Each section has been assigned jurisdiction over pertinent groups of test procedures of Committee C-8's literature. A survey has been made of apparatus and equipment involved in each of the Committee C-8 test methods to learn whether adequate description of the equipment is presented. Pertinent subject matter handled by each section of the committee is as follows:

Section A on Load (J. D. Sullivan, chairman).—Active work has been carried out by the section in the way of obtaining a cross-section of the requirements and ramifications of the hot-load test for various types of refractories as conducted by laboratories throughout the country. The response was useful in the study which lead to the worthwhile revision of Method C 16 embodied in the tentative recommendations presented earlier in the report. Method C 133 for crushing and modulus of rupture of refractories was brought up-to-date by incorporating the rate of load application tolerance mentioned, and consideration has been given to the possibility of incorporating a procedure for hot modulus of rupture of refractories but it appears that this might well be delayed until later, pending further developments.

Section B on Spalling (R. E. Birch, chairman) provided a new paragraph for the four spalling procedures, describing more completely the operation involved in cleaning the test brick after spalling, and this has been recommended for adoption. In the present spalling procedure relating to fireclay plastic refractories, the method is to weigh the test brick after spalling and after the removal of the spalls, a practice which could be improved. Consideration is being given to an alternate procedure which involves making use of the ignition loss of the unfired test brick in calculating the spalling loss from the original weight of the specimens and after testing and the removal of the spalls. A recommended practice presenting more detailed information than is thought necessary for incorporation in the actual spalling procedures is to become a part of the forthcoming Manual of A.S.T.M. Standards on Refractories.

Section C on Temperature (J. L. Carruthers, chairman).—A study is being carried out in the section to learn of the effect of small variations in size of standard pyrometric cones on the end point. Exploratory work on this subject indicates that the size effect is not of real consequence. The subject is of particular interest in the P.C.E. test, especially as relating to the size of the molded test cones where variation is likely to enter because of mold wear, technique, and drying and firing shrinkage of the material to be tested.

Further information is being collected on the long-standing problem of P.C.E. determinations as made in oxyacetylene, gas-air, and electric furnaces. A fellowship at Rutgers University is studying the matter and the committee expects that in the not too distant future, a specific type of furnace for the P.C.E. test will become a part of the C-8 procedure.

Section E on Analysis (L. J. Trostel, chairman) has recommended as an editorial change the inclusion in the analytical methods C 18 the use of an 0.2 *N* solution of potassium bichromate together with an appropriate indicator. This provides latitude in the method for those who may want to titrate with the bichromate rather than potassium permanganate. Efforts are being made to have the Bureau of Standards prepare a better suited standard sample of silica brick, which would have an alumina content of the order of 1 per cent rather than the 1.96 per cent of the present standard, which is too high to represent the present-day product.

Section F on Tests on Refractory Insulation (W. R. Kerr, chairman) has reported exploratory work on the effect of the size of insulating fire brick test specimens when used in the permanent linear change test. This indicates that pieces smaller than a 9-in. brick may give reliable data and be quite satisfactory for plant control work where the size of specimen in quantity testing would be of consequence. Trials were reported on the effect of gas atmospheres on insulating fire brick in the permanent shrinkage and hot-load tests.

Section G on Porosity and Permanent Volume Change (J. F. Kelly, chairman, replacing C. E. Fulton) is giving thought to better specifications for the properties of supporting brick for test specimens in the permanent linear change test Method C 113. The present procedure calls for brick from the same lot to be used as a supporting member for the specimens, or that they should be of at least equal refractoriness, the latter being an inadequate statement. Experimental work to date indicates that such brick can be selected on the basis of their linear change in the test rather than "refractoriness." Another consideration on the committee's program relates to the matter of making use of volume change as well as linear change in reporting the behavior of the reheated brick. The section has been requested to make a survey of the literature to learn of methods now in use for determining the permeability of refractories at room and furnace temperatures, and this eventually may lead to a procedure for determining permeability.

Section H on Mortars and Plastic Refractories (R. S. Bradley, chairman) has recommended that there be incorporated in the Method of Test for the Refractoriness of Air-Setting Refractory Mortars (C 199) a description of a suitable oven, which has been overlooked. This editorial change would mean the inclusion of a paragraph essentially the same as Paragraph 2(*c*) in Method C 198. Another editorial change relating to that method would be the insertion of the word "respective" after "the following" in Paragraph 2(*a*) so as to clarify the grade of brick to be used in carrying out the test on different classes of mortars.

Subcommittee IV on Heat Transfer (C. L. Norton, Jr., chairman, replacing R. H. Heilman) is making a critical review of the manuscript prepared by the chairman, which presents detailed information for conducting thermal conductivity tests embodied in Methods C 182, 201, and 202. The purpose of this is to condense the information to the extent that it would be suitable for incorporating in the new Manual on Refractories. The subcommittee has requested and encouraged work at the Bureau of Standards on the fundamentals of procedure and equipment for determining the thermal conductivity of high-temperature insulating materials. One result of this endeavor is the report made to Committee C-8 by H. C. Dickinson of the Heat Transfer Section of the Bureau, in which he outlined what appears to be a promising rapid method for at least the control testing of insulating fire brick to obtain the *K* factor. The test is in its early stages of development and the subcommittee will follow its progress with interest.

Subcommittee V on Precision and Tolerance (W. S. Debenham, chairman, replacing R. A. Heindl).—Since the preparation of the tables incorporated in four of the committee's specifications for the test specimen requirements, the subcommittee has not been in a position to report new subject matter.

Subcommittee VI on Nomenclature (R. A. Heindl, chairman) has been active in originating and revising definitions for refractories, and the result of their work is evidenced by the committee's adoption of five new definitions and the revision of the definitions for "burning of refractories" and "fire clay." Additional definitions under study pertain to magnesite-chrome brick and chrome-magnesite brick.

Subcommittee VII on Specifications (G. A. Bole, chairman) has made recommendations relating to low heat duty fireclay brick involving the increase in the modulus of rupture requirements as given in Specifications C 64, C 106, and C 153. It has also presented new specifications for publication as tentative on the subject of castable refractories for use in boiler furnaces and incinerators. This helps to round out the field covered by Committee C-8 specifications and should prove to be a useful addition. The subcommittee is devoting attention to the desirability of incorporating in Specifications C 105 for Ground Fire Clay as a Mortar for Laying-Up Fireclay Brick requirements for clay used as a daubing and mudding mix or of the possibility of developing a separate specification covering the last two uses.

Subcommittee VIII—Editorial (L. J. Trostel, chairman) has been particularly active in reviewing all of the Committee C-8 literature preparatory to the publication of a new edition of the Manual of A.S.T.M. Standards on Refractory Materials as issued by the Society. Even though a number of significant editorial changes have been made along with routine work, mention of these will not be made here. It is expected that the new manual will be published late in the summer, and at that time there can be incorporated in it the new subject matter presented in this report forformal action by the Society.

Subcommittee IX on Classifications (S. M. Swain, chairman) has made a critical review of the Classification of Fireclay Refractories (C 27 – 41) since no changes have been made. This resulted, however, in a reaffirmation by the committee of the classification as it now stands. Recently requests have been made to incorporate in the classification statements pertaining to what are known as double-burned super duty fireclay brick. This will be reported on at a later time.

Subcommittee X on Petrography (H. M. Kraner, chairman).—The chairman of the subcommittee has prepared a manuscript entitled "Petrographic Techniques Applied to Refractories," which presents descriptive matter relating to thin section, powder, and polished surface techniques, and quantitative microscopic analysis, as well as a bibliography on these subjects. This subject matter will also be incorporated in the new manual.

Subcommittee XI on Special Refractories (M. C. Booze, chairman) is reviewing the requirements involved in the preparation of specifications and test procedures relating to special refractory materials, a field which is new to Committee C-8. Because of the growing importance of this group of refractory materials, specifications and test methods relating to them would be a worthwhile contribution.

This report has been submitted to letter ballot of the committee, which consists of 33 members; 30 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. D. SULLIVAN,
Chairman.

L. J. TROSTEL,
Vice-Chairman.

S. M. PHELPS,
Secretary.

REPORT OF COMMITTEE C-9*

ON

CONCRETE AND CONCRETE AGGREGATES

Committee C-9 on Concrete and Concrete Aggregates has had a year of accelerated activity which has done much to compensate for its reduced productivity during the war years. Three meetings have been held: One on June 27, 1946, during the Annual Meeting of the Society in Buffalo, one in Washington, D. C., on December 3, 1946, and one in Cincinnati on March 28, 1947. While a considerable amount of work has been accomplished, a principal result of the renewed activity has been to get new work under way.

The reorganization in the committee referred to in the 1946 Report will be completed at the Annual Meeting of the Society in June, 1947. It involves the division of the committee activities into three groups: Administration, Research, and Specifications and Test Methods. Subcommittees will be made smaller than at present, and the scope of work assigned to each will be made narrower and more specific. With the close of the Annual Meeting, existing subcommittees are to be discharged and the newly appointed subcommittees will take over their work. Of course, when existing subcommittees fit into the new pattern they will be reappointed.

In 1946 the committee suffered the loss, through death, of one of its younger and more active and productive members, William F. Kellermann. Mr. Kellermann, a Senior Materials Engineer of the Public Roads Administration, had been a member of the committee since 1938. While he contributed to many phases of the committee's work, his principal activity was that of chairman of Subcommittee XIII on Curing of Concrete.

Sanford E. Thompson Award.—The Sanford E. Thompson Award for a paper of outstanding merit on concrete and concrete aggregates presented to the Society will be made this year to Mr. William Lerch, Research Chemist, Research Laboratories, Portland Cement Assn., for his paper on "The Influence of Gypsum on the Hydration and Properties of Portland Cement Pastes," published in the 1946 *Proceedings*.[1] This is the sixth Sanford E. Thompson Award, previous ones having been made in 1946, 1944, 1941, 1940, and 1939.

The Regulations governing Committee C-9 were revised during the year with provision being made for election to honorary membership of "members in good standing who have participated actively in the work of the committee for a period of 20 years." In accordance with these new Regulations the committee has recognized the long and valuable services of P. J. Freeman, F. R. McMillan and L. W. Walters by electing them as honorary members.

I. Revision of Tentative

Tentative Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192-

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1252 (1946).

44 T).[2]—The Committee recommends the following revision in this method:

Section 4 (*c*).—Change to read as follows:

4.(*c*) *Machine Mixing.*—The procedure specified for hand mixing shall be followed unless a different procedure is better adapted to the mixer being used. Precautions shall be taken to compensate for mortar retained by the mixer so that the finished batch as used will be correctly proportioned. To eliminate segregation, machine mixed concrete shall be deposited in a water-tight, clean, damp metal pan and remixed by shovel or trowel.

NOTE.—It is difficult to recover all of the mortar from certain types of mixers, particularly those of the revolving drum type, and when such conditions obtain, one of the following procedures is suggested to insure the correct final proportions in the batch:

(*1*) *"Buttering the Mixer."*—Just prior to mixing the test batch, the mixer should be "buttered" by mixing a batch proportioned to simulate closely the test batch. The mortar adhering to the mixer after discharging is intended to prevent loss of mortar from the test batch. The discharged test batch may be adjusted to proper weight by the addition or subtraction of mortar.

(*2*) *"Over Mortaring" the Mix.*—The test mix may be proportioned by the use of an excess of mortar to compensate for that which, on the average, adheres to the mixer. In this case the mixer is cleaned before mixing the test batch.

II. ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Method of Test for Surface Moisture in Fine Aggregate (C 70 – 44 T)[2] be approved for reference to letter ballot of the Society for adoption as standard, without revision.

III. REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption, revisions in two of its standards, as set forth below, and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society.

Standard Method of Test for Measuring Mortar-Making Properties of Fine Aggregate (**C 87 – 46**):[2]

New Section.—Add a new Section 8 to read as follows:

8. *Testing Specimens.*—Specimens, including cylinders, shall be tested for compressive strength in accordance with the provisions prescribed in Section 12 of the Standard Method of Test for Compressive Strength of Hydraulic-Cement Mortars (A.S.T.M. Designation: C 109).[2]

Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (**C 131 – 46**).[2]—This method is under the joint jurisdiction of Committee D-4 on Road and Paving Materials and Committee C-9. The following revisions, substantially editorial have been approved by Committee D-4 and it is confidently expected that they will be concurred in by Committee C-9 at its meeting in June:

Section 2 (*b*).—Change to read as follows:

(*b*) *Sieves.*—All sieves herein specified for use shall conform to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11).

Section 4.—Change the first sentence to read as follows: "The test sample shall consist of 5000 g. of clean aggregate which has been dried in an oven at 105 to 110 C. (221 to 230 F.) substantially to constant weight and shall conform to one of the gradings shown in Table I."

Section 5.—Change the last two sentences to read as follows:

At the completion of the test, the material shall be discharged from the machine and a preliminary separation of the sample made on a sieve coarser than the No. 12 (1680-micron). The finer portion shall then be sieved on a No. 12 sieve in a manner conforming to Section 5 (*a*) on the Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136). The material coarser than the No. 12 sieve shall be washed, dried in an oven at 105 to 110 C. (221 to 230 F.) to substantially constant weight, and accurately weighed to the nearest gram (Note).

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[2] 1946 Book of A.S.T.M. Standards, Part II.

IV. TENTATIVE REVISION OF STANDARD

Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (**C 131 - 46**).[2]—The committee recommends the following changes as a tentative revision of this method:

Section 3 (*a*).—Change the first sentence to read as follows: "The abrasive charge, when required, shall consist of cast-iron spheres or steel spheres approximately 1⅞ in. in diameter and each weighing between 390 and 445 g."

Section 3 (*b*).—Change this section to read as follows:

(*b*) The abrasive charge, depending upon the grading of the test sample as described in Section 4, shall be as follows:

Grading	Number of Spheres	Weight of Charge, g.
A	12	5000 ± 25
B	11	4584 ± 25
C	8	3330 ± 20
D	6	2500 ± 15
E	none	....
F	none	....
G	none	....

Section 4.—Change to read as follows:

4. *Test Sample.*—The test sample shall consist of clean aggregate which has been dried in an oven at 105 to 110 C. (221 to 230 F.) to substantially constant weight and shall conform to one of the gradings shown in Table I (Note). The grading or gradings used shall be those most nearly representing the aggregate furnished for the work.

Table I.—Change to read as follows:

TABLE I.—GRADINGS OF TEST SAMPLES.

Sieve Size (Square Openings)		Weight and Grading of Test Sample, g.						
Passing	Retained On	A	B	C	D	E	F	G
3 in.	2 in.	....	....	....	....	5000[a]	....	....
2 in.	1½ in.	....	....	....	....	5000[a]	5000[a]	....
1½ in.	1 in.	1250	....	....	....	....	5000[a]	5000[a]
1 in.	¾ in.	1250	....	....	....	....	....	5000[a]
¾ in.	½ in.	1250	2500	....	....	....	....	....
½ in.	⅜ in.	1250	2500	....	....	....	....	....
⅜ in.	No. 3	....	....	2500	....	....	....	....
No. 3	No. 4	....	....	2500	....	....	....	....
No. 4	No. 8	....	....	....	5000	....	....	....

[a] Tolerance of plus or minus 2 per cent permitted.

NOTE.—It is recognized that different specification limits will be required for gradings E, F, and G than for A, B, C, and D. It is urged that investigations be conducted to determine the relationship, if any, which exists between results for these gradings and those tested with an abrasive charge. It is also urged that modifications of the specified procedure be investigated and it is specifically recommended that data be developed for gradings E, F, and G using weights of sample of 5000 and 10,000 g., an abrasive charge of twelve 1⅞ in. cast-iron or steel spheres, and 500 revolutions of the machine.

Section 5.—Change to read as follows:

5. *Procedure.*—The test sample and the abrasive charge, if any, shall be placed in the Los Angeles abrasion testing machine and the machine rotated at a speed of from 30 to 33 rpm. For gradings A, B, C, and D (Section 4) the machine shall be rotated for 500 revolutions; for gradings E, F, and G it shall be rotated for 1000 revolutions. The machine shall be so driven and so counterbalanced as to maintain a substantially uniform peripheral speed. If an angle is used as the shelf, the machine shall be rotated in such a direction that the charge is caught on the outside surface of the angle. At the completion of the test, the material shall be discharged from the machine and a preliminary separation of the sample made on a sieve coarser than the No. 12. The finer portion shall then be sieved on a No. 12 (1680-micron) sieve as prescribed in Section 5(*a*) of the Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (A.S.T.M. Designation: C 136). The material coarser than the No. 12 sieve shall be washed, dried in an oven at 105 to 110 C. (221 to 230 F.) to substantially constant weight, and accurately weighed to the nearest gram (Note).

NOTE.—Attention is called to the fact that valuable information concerning the uniformity of the sample under test may be obtained by determining the loss after 100 revolutions. When this determination is made, care should be taken to avoid loss of any part of the sample; the entire sample, including the dust of abrasion, shall be returned to the testing machine for the completion of the test.

V. REVISION OF STANDARD AND REVERSION TO TENTATIVE

Standard Specifications for Ready Mixed Concrete (**C 94 - 44**).[2]—The committee recommends that these specifications be revised, as appended hereto,[3] and reverted to tentative.

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[3] These revised specifications were accepted by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part II.

VI. NEW TENTATIVE

The committee recommends for publication as tentative the proposed new Method of Test for Fundamental Transverse Frequency of Concrete Specimens for Computing Modulus of Elasticity (Sonic Method).[4]

VII. CONTINUATION OF TENTATIVES WITHOUT REVISION

Tentative Method of Test for Water Retention Efficiency of Methods of Curing Concrete (**C 156 – 44 T**), and *Tentative Specifications for Waterproof Paper for Curing Concrete* (**C 171 – 42 T**).—The subcommittee having jurisdiction over these methods and specifications has been inactive due to the death of its chairman. Therefore, until this subcommittee is again active, it is recommended by the committee that they be retained as tentative.

Tentative Method of Test for Air Content (Volumetric) of Freshly Mixed Concrete (**C 173 – 42 T**).—The committee feels it inadvisable to adopt as standard at the present time this method of test for air content of fresh concrete due to the work now being actively conducted in this field.

VIII. NEW METHODS IN PREPARATION

The committee has prepared a Proposed Tentative Method of Evaluation of Air-Entraining Admixtures for Concrete for publication as information in the ASTM BULLETIN[5] with the expectation of submitting it at a later date to the Administrative Committee on Standards for appropriate action, after revision in the light of such comments as are received.

The recommendations appearing in this report have been approved by letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

This report has been submitted to letter ballot of the committee, which consists of 73 voting members; 68 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

K. B. WOODS,
Chairman.

STANTON WALKER,
Secretary.

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[4] This method was accepted as tentative by the Society and appears in the 1947 Supplement to Book of A.S.T.M. Standards, Part II.

[5] ASTM BULLETIN, No. 148, October, 1947, p. 29.

[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

REPORT OF COMMITTEE C-14*
ON
GLASS AND GLASS PRODUCTS

Committee C-14 on Glass and Glass Products held one meeting during the year, in Atlantic City, N. J., on April 24, 1947.

A proposed revision of the By-laws of Committee C-14 to provide for the membership of the Advisory Committee to consist of the three officers and the chairmen of the subcommittees has been referred to letter ballot of the committee.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Definition of the Term Glass (C 162 - 45 T) be approved for reference to letter ballot of the Society for adoption as standard without revision. This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (H. H. Holscher, chairman) is preparing a glossary of terms used in the glass industry.

Subcommittee II on Chemical Analysis (G. E. F. Lundell, chairman).—Arrangements are in progress whereby a new standard sample of glass sand of low-iron content (approximately 0.02 per cent) will be prepared and issued by the National Bureau of Standards. This sand will be furnished by F. C. Flint of Hazel-Atlas Glass Co. and analyzed at the Bureau of Standards and in the laboratories of the members of Subcommittee II. The analysis will include determination of Fe_2O_3, Al_2O_3, TiO_2, ZrO_2, Cr_2O_3, CaO, and MgO. It is hoped that the new standard will be ready for distribution before the end of the year.

Progress on the formulation of standard methods for the chemical analysis of glass-making materials such as feldspar, fluorspar, and lime has been slow, and no recommendations can be made at this time.

Subcommittee III on Chemical Properties (J. F. Greene, chairman).—The last report submitted to Committee C-14 on methods for testing durability of glass and glass containers has been the object of study by Subcommittee VII-A on Pharmaceutical Containers, with the aim of selecting methods suitable for recommendation to the U. S. Pharmacopoeia. Two series of comparative tests have been run by Subcommittee VII-A with the cooperation of Subcommittee III. A third series is being arranged. It is expected that the methods finally agreed upon will be found useful by Subcommittee III.

Subcommittee VII on Glass Containers (K. C. Lyon, Chairman).—There have been no formal activities of this subcommittee since the last report. Several of the members have held informal discussion groups at different times, discussing changes which may need to be made in A.S.T.M. Methods C 147, C 148, and C 149. There have also been some suggestions that other test methods, in addition to the above, which are generally applied to glass containers, should be standardized. These discussion groups have been advised to submit their suggestions for consideration of the committee.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at A S.T.M. Headquarters.

Subcommittee VII-A on Pharmaceutical Containers (F. C. Flint, chairman).—The second set of bottles was tested for chemical attack, using procedures derived from the ASTM methods submitted in 1943 in conjunction with the U.S.P. methods according to the Twelfth Revision. Details of procedure were gone over and three methods chosen. These are to be checked on another series of tests by the subcommittee, after which they will be offered to Committee C-14 for approval as tentative.

Subcommittee VIII on Flat Glass (D. E. Sharp, chairman).—Early in 1946, steps were taken by members of the staff of the Bureau of Standards toward revision of Federal Specification DD-G-451 for Glass: Flat, for Glazing Purposes. Copies of the proposed revision have been distributed among the membership of the subcommittee. A number of the members submitted suggestions for correction of the first draft, the suggestions being sent directly to the Bureau of Standards with copies being sent to the subcommittee also. There has been no circulation of the data within the committee.

A further revision of the proposed specification was in progress in 1946 by the Technical Committee on Glassware, Chinaware, etc. of the Federal Specification Board. This work was not completed in 1946 but the proposed revision became available early in 1947 and copies have been circulated to the membership of Committee C-14. The matter may be said to be of current interest but the status is such that activity in this direction will be reported upon at a later date.

One of the members of Subcommittee VIII is acting as representative of A.S.T.M. on a sectional committee functioning under the American Standards Association which deals with specifications for safety glass, and which has been active in 1946.

This report has been submitted to letter ballot of the committee, which consists of 49 members; 30 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

LOUIS NAVIAS,
Chairman.

S. R. SCHOLES,
Secretary.

REPORT OF COMMITTEE C-16*

ON

THERMAL INSULATING MATERIALS

Committee C-16 on Thermal Insulating Materials and its subcommittees held meetings during the past year on two occasions: October 21 to 23, 1946, and February 24 to 26, 1947. These meetings were well attended and much interest was evidenced.

I. New Tentative

The committee recommends that the proposed new Method of Test for Water Vapor Permeability of Sheet Materials Used in Connection with Thermal Insulation[1] be accepted for publication as tentative.

This method of test is similar to the Tentative Method of Test for Water Vapor Permeability of Paper and Paperboard (D 783 - 44 T) but because of its connection with thermal insulation it differs in scope, in the units specified for reporting, and in some other details. It does not specify temperature and humidity conditions as does Method D 783, since the committee recognized a frequent need for measurements at more than one set of conditions.

II. Revision of Standard, Immediate Adoption

The committee recommends revision of the Standard Definitions of Terms Relating to Thermal Insulating Materials (C 168 - 44) by the addition of six definitions of the following terms: bulk density of thermal insulating cement, dry covering capacity of thermal insulating cement, thermal conductance, thermal conductivity, volume change upon drying of thermal insulating cement, and wet covering capacity of thermal insulating cement, as appended hereto.[2] The committee accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society.

III. Tentative Revision of Standard

Standard Definitions of Terms Relating to Thermal Insulating Materials (**C 168-44**).—The committee recommends the following definition for publication as a tentative revision of this standard:

Molded-Type Pipe Insulation.—A rigid or semi rigid thermal insulating material preformed in semi-cylindrical or segmental sections or both, usually 3 ft. in length, for application on pipe or tubing, furnished with or without finish or attachments.

IV. Revisions of Tentatives

The committee recommends that the following three tentatives be revised as follows:

Tentative Specifications for Mineral Wool Thermal Insulating Cement (**C 195 - 45 T**):[3]

Section 1.—Change "These specifications cover thermal insulating material composed predominantly of mineral wool . . ." to read "These specifications cover mineral wool thermal insulating material. . . ."

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] This method was accepted as tentative by the Society and appears in the 1947 Supplement to Book of A.S.T.M. Standards, Part II.

[2] These revised definitions were accepted by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part II.

[3] 1946 Book of A.S.T.M. Standards, Part II.

New Section.—Add a new Section 2 to read as follows and renumber the subsequent sections accordingly: "2. *Composition.*—The cement shall be composed predominantly, by weight, of mineral wool, with a suitable proportion of heat-resistant binder."

Tentative Specifications for Expanded or Exfoliated Vermiculite Thermal Insulating Cement (**C 196 - 44 T**):[3]

Section 1.—Change "These specifications cover thermal insulating material composed predominantly of expanded or exfoliated vermiculite . . ." to read "These specifications cover expanded or exfoliated vermiculite thermal insulating material . . ."

New Section.—Add a new Section 2 to read as follows and renumber the subsequent sections accordingly: "2. *Composition.*—The cement shall be composed predominantly, by weight, of expanded or exfoliated vermiculite, with a suitable proportion of heat-resistant binder."

Tentative Specifications for Diatomaceous Silica Thermal Insulating Cement (**C 197 - 44 T**):[3]

Section 1.—Change "These specifications cover thermal insulating material composed predominantly of diatomaceous silica . . ." to read "These specifications cover diatomaceous silica thermal insulating material."

New section.—Add a new Section 2 to read as follows and renumber the subsequent sections accordingly: "2. *Composition.*—The cement shall be composed predominantly, by weight, of diatomaceous silica, with a suitable proportion of heat-resistant binder."

The recommendations appearing in this report have been approved by letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

V. CONTINUATION OF TENTATIVES WITHOUT REVISION

The committee recommends that the following tentatives which have been published for two years or more be continued as tentative, since active work on revision is under way but not completed.

Tentative Specifications for 85 per cent Magnesia Thermal Insulating Cement (C 193 - 44 T),

Tentative Specifications for Asbestos Thermal Insulating Cement (C 194 - 44 T), and

Tentative Method of Test for Flexural Strength of Preformed Block Type Thermal Insulation (C 203 - 45 T).

This report has been submitted to letter ballot of the committee which consists of 55 members; 50 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

RAY THOMAS,
Chairman.

K. M. RITCHIE,
Secretary.

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[4] The letter ballot vote on these recommendations was favorable, the results of the vote are on record at A.S.T.M. Headquarters.

REPORT OF COMMITTEE C-18*
ON
NATURAL BUILDING STONES

Committee C-18 on Natural Building Stones held a meeting in Washington, D. C., on March 3, 1947, and Subcommittees II on Nomenclature, IV on Specifications, and V on Research held meetings at the same time. Four meetings of the Advisory Subcommittee were held in Washington, D. C., during the year.

One resignation has occurred during the year and 5 new members have been added. The membership now consists of 21 members, of whom 10 are producers, 6 consumers, and 5 as general interest members.

New problems which have been presented to the committee include (*1*) a means of selecting durable stone for use as rip-rap, requested by the U. S. Reclamation Service, and (*2*) specifications for monumental marble, requested by the American Battle Monuments Commission. It was decided that rip-rap stone is not within the scope of this committee but that monumental stones of all types should be considered as building stones. Durability tests will be given serious consideration.

The committee has been asked to cooperate with the American Standard Association in formulating specifications for building granite.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the Standard Methods of Test for Absorption and Apparent Specific Gravity of Natural Building Stone (C 97 - 36), and the Standard Method of Flexure Testing of Natural Building Stone (Determination of Modulus of Rupture) (C 99 - 36), as appended hereto,[1] and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

These recommendations have been submitted to letter ballot[2] of the committee, the results of which will be reported at the Annual Meeting.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Nomenclature (L. W. Currier, chairman) has given consideration to definitions of various types of stone. It was considered that it would be necessary to divide many of these into two categories, namely, scientific definitions to describe them adequately and commercial definitions of a less restrictive nature. Considerable progress has been made along this line and a list of definitions has been submitted to the committee for comment. This list includes definitions of granite, alkalic feldspars, commercial (building) granite, gneiss, "black granite," limestone, calcite limestone, magnesian (Dolomitic) limestone, dolomite, travertine, marble, commercial marble, calcite marble, magnesian (Dolomitic) marble, dolomite marble, onyx (Mexican) marble, serpentine marble, ophicalcite marble, verd antique, greenstone marble,

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*** Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.**

[1] These revised methods were accepted by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part II.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

sandstone, quartzite, conglomerate, freestone, flagstone, bluestone, and brown stone. It is expected that these definitions will be proposed for publication as tentative during the coming year.

Subcommittee III on Methods of Testing (D. W. Kessler, chairman) has held no meetings during the year but by conferences and correspondence it has prepared the revisions recommended for immediate adoption in the two standard test methods, referred to earlier in this report, which have stood for several years without change. Revisions in two other Standard Methods: Flexure Testing of Slate (Modulus of Rupture, Modulus of Elasticity) (C 120 - 31) and Test for Water Absorption of Slate (C 121 - 31) are under consideration.

Subcommittee IV on Specifications (W. C. Clark, chairman) has given consideration to the properties of stone that should be made the bases of specifications. Progress has been made along the line of preparing specifications for roofing slate and this subcommittee expects to submit a proposal for this type of stone during the coming year.

Subcommittee V on Research (J. R. Shank, chairman) has discussed various problems which require study. One of these includes the development of a suitable test for determining the toughness of slate. The object is to find a means of selecting slate that will be less subject to breakage on roofs. Another problem relates to the evaluation of polished surfaces and determining the degree of polish on different materials.

A cooperative study of different phases of weathering involving an exposure test wall was started in 1941 by the National Bureau of Standards and the Society but this group remained inactive during the war period. This project has been resumed and a design has been worked out which will include about 2000 samples of stone so oriented that each may be located and identified by its position. Thirty different types of stone and many varieties of the important types will be represented. The generous response of the producers to a request for larger blocks for the base, coping and quoins indicates that the wall may be completed during the present year.

This is a long-range project that is expected to give some valuable information on the relation between laboratory tests and actual weathering. One-half of the wall is to be set with portland-cement mortar and the other half with lime mortar. Certain combinations of types, which have been reported to be detrimental, are to be made. This involves placing a large area of calcareous stones over siliceous materials and a considerable area of magnesian stones over calcareous types. Incidentally it is expected to find which stones retain their original colors over a long period of time and which change color either from weathering or are affected by discolorations not attributable to weathering.

This report has been submitted to letter ballot of the committee, which consists of 21 members; 18 members returned their ballots, of whom 17 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

OLIVER BOWLES,
Chairman.

D. W. KESSLER,
Secretary.

REPORT OF COMMITTEE D-1*
ON
PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year in Buffalo, N. Y., on June 26, 1946, in connection with the Annual Meeting of the Society, and in Philadelphia, Pa., on February 26, 1947, in connection with the Spring Group Committee Meetings.

During the past year a new Subcommittee on Resins has been organized to undertake the development of methods of test and specifications for resins.

The committee arranged an all day technical program in connection with its meeting, on June 26, 1946.

The committee also arranged the program for a Symposium on Paint and Paint Materials[1] which was held under the cosponsorship of Committee D-1 and the Philadelphia District Council in Philadelphia, Pa., on February 25, 1947.

The D-1 Membership Committee, which was organized in 1946 to obtain new members for Committee D-1, particularly members engaged directly in the protective coating industry, has been very active. Since June, 1946, the Membership Committee has succeeded in adding 30 new members to Committee D-1. It is of particular interest that 24 of these were directly connected with the manufacture of paints. This represents a rather large increase in the percentage of D-1 members in this classification.

The committee is considering the most suitable locations for test sites for exposure tests of protective organic coatings. The committee expects to get more information on the present A.S.T.M. sites and to select sites not simply on the basis of geographical location, but on the basis of the specific weather conditions and other pertinent qualifications. Recommendation will be made to the Society shortly after July, 1947.

The D-1 Inter-committee Relations Committee which is composed of the D-1 representatives on other technical committees is closely following the activities of other technical committees which may be of interest to Committee D-1.

Subsequent to the 1946 Annual Meeting, Committee D-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:

Test for Changes in Protective Properties of Organic Coatings on Steel Surfaces when Subjected to Immersion (D 870 – 46 T) (Formerly ES – 35),

Revision of Tentative Method of:

Preparation of Steel Panels for Testing Paint, Varnish, Lacquer and Related Products (D 609 – 46 T),

Test for Evaluating Degree of Settling of Traffic Paint (D 869 – 46 T),

Revision of Tentative Recommended Practice for:

Operating Light and Water Exposure Apparatus (Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products (D 822 – 46 T), and

Tentative Revision of Standard Specifications for:

C. P. Zinc Yellow (Zinc Chromate) (D 478 – 41).

These recommendations were accepted by the Standards Committee on September 9, 1946, and the new and revised tentatives together with the tentative revision of D 478 appear in the 1946 Book of A.S.T.M. Standards, Part II.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Issued as a separate publication, 1947.

The following specifications under the jurisdiction of Committee D-1, previously approved by the American Standards Association as American Standard, have been reapproved in their revised form. The date of approval is December 17, 1946:

Standard Specifications for:

Lampblack (ASTM D 209 - 46; ASA K26.1—1946),
Bone Black (ASTM D 210 - 46; ASA K36.1—1946),
Prussian Blue (ASTM D 261 - 46; ASA K29.1—1946), and
Chromium Oxide Green (ASTM D 263 - 46; ASA K37.1—1946).

RECOMMENDATIONS AFFECTING STANDARDS

Fifteen subcommittees have submitted reports of progress, twelve of which cover recommendations affecting standards as a result of which the committee is submitting three new tentatives, revisions of three tentatives, one tentative revision of a standard, revisions of seventeen standards for immediate adoption, and is recommending the adoption of three tentatives as standard without revision and one with revision, the adoption as standard of three tentative revisions of standards, the withdrawal of two standards, and the revision of two standards and reversion to tentative.

These recommendations, which are given in detail under the activities of subcommittees, have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1a]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Drying Oils (Francis Scofield, chairman) recommends a revision for immediate adoption of the Standard Specifications for Perilla Oil, Raw or **Refined (D 125 - 41).**[2] This revision consists of changing from the Hanus to the Wijs method for determining iodine number and changing the requirements for the minimum iodine number from "191" to read "196."

The subcommittee also recommends a revision for immediate adoption of Standard Methods of Testing Drying Oils (D 555 - 41)[2] to consist of the following changes to clarify the method and bring it up to date:

Section 2.—At the end of Paragraph (*a*), add the following sentence: "Standard potassium hydroxide solution is made in the same manner, using a stoichiometrically equal amount of KOH."

At the end of Paragraph (*b*), add the following sentence: "Alcoholic potassium hydroxide solution is made in the same manner, using a stoichiometrically equal amount of KOH."

Change Paragraph (*d*) to read as follows: "(*d*) *Wijs Iodine Monochloride Solution.*—Prepare in accordance with Section 12 (*b*) of the Standard Methods of Sampling and Analysis of Shellac (A.S.T.M. Designation: D 29)."

Delete the description of the Hanus Solution in Paragraph (*e*) and reletter the remaining paragraphs accordingly.

Change Paragraph (*f*) to read as follows: "(*g*) Chloroform, U.S.P.—When titrated to the iodine starch end point, shall show no return of color on standing for 1 min."

At the end of the third sentence of Paragraph (*i*), add "or National Bureau of Standards standard sample No. 136 of potassium dichromate."

Section 4.—Change to read as follows: "Use a pycnometer, accurately standardized at 15.5 C. and having a capacity of at least 25 ml., or any other equally accurate method. Make the test at any convenient temperature, between 15 and 30 C., and correct the result to 15.5 C. by adding 0.00064 for each degree Centigrade above 15.5 C."

[1a] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

[2] 1946 Book of A.S.T.M. Standards, Part II.

Section 5.—Change the title to read "*Acid Number.*"

Delete the phrase "To determine the acid number of boiled or raw linseed oil and of raw tung oil" from the first part of the first sentence so that the sentence now begins "Weigh from 5 to 10 g. of the sample. . . "

Section 6.—Delete this section and renumber the subsequent sections accordingly.

Section 9.—Reletter as eight and in the third sentence change the amount of oil from "0.09 to 0.15" to read "0.11 to 0.13."

Section 10.—Delete this section on "Iodine Number, Hanus."

Section 11.—Change the first sentence to read as follows: "Place 10 g. of the sample in an accurately weighed 50-ml. flask and weigh."

Section 12.—Change to read as follows: "Transfer a portion of the sample to a clear glass tube and note the appearance. If the oil is cloudy when cold, heat to 65 C. for 5 min. and again note the appearance."

Section 13.—Change to read as follows: "Determine the color in accordance with the procedure described in Sections 5 to 7 of the Standard Methods of Testing Varnishes (A.S.T.M. Designation: D 154)."[3]

The subcommittee is working on the development of new testing methods for modified and synthetic drying oils. It is developing a heat bodying test and a method for determining acetone number of bodied oils. In the cooperative work good agreement was obtained by the six laboratories in the heat bodying tests and there is sufficient background to write up the method. In the work on acetone number, variations have been encountered and further work will be required.

The subcommittee has reviewed the various A.S.T.M. specifications for drying oils for possible revisions. Work is being continued on developing specifications for dehydrated castor oil.

Subcommittee III on Bituminous Emulsions (E. M. Berger, chairman) has been preparing specifications for asphalt-base emulsions and coal-tar base emulsions for use as protective coatings for metal. The subcommittee has also been preparing methods of testing bituminous emulsions intended for use as protective coatings for metals.

The subcommittee expects to study new methods for stability and water resistance of bituminous emulsions during the coming year.

Subcommittee IV on Traffic Paints (W. G. Vannoy, chairman) recommends a new Tentative Method of Evaluating Degree of Resistance of Traffic Paint to Chipping.[4]

The subcommittee also recommends that the Tentative Method of Evaluating Degree of Resistance of Traffic Paint to Abrasion, Erosion, or a Combination of Both in Road Service Tests (D 821 - 45 T) be adopted as standard.

The subcommittee recommends for immediate adoption the following revision of Standard Method of Test for Light Sensitivity of Traffic Paint (D 712 - 46):[2]

Section 3(a) and (b).—In the first sentence of each paragraph change "enclosed type carbon arc light" to read "carbon-arc light source."

At the end of Paragraph (*b*) add the following: "The test paint shall not show greater loss in brightness nor greater discoloration than mutually agreed upon by the purchaser and the seller for a specific paint. The type of arc shall be agreed upon."

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[3] 1947 Supplement to Book of A.S.T.M. Standards, Part II.

[4] These methods and specifications were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part II.

The subcommittee has been investigating the use of a portable instrument for measuring night visibility of traffic paints and work is being continued on accelerated tests for durability and for degree of settling. The subcommittee expects to recommend a method of test for evaluating the degree of resistance of traffic paints to bleeding.

Subcommittee V on Volatile Hydrocarbon Solvents for Organic Protective Coatings (M. B. Chittick, chairman) is planning a program of cooperative testing to establish a correlation between analine point and Kauri butanol numbers.

The subcommittee is also initiating a program to define solvency in terms of viscosity reduction and dilution limits and to standardize by means of cooperative testing a procedure for determining relative solvency based on viscosity ratios. The subcommittee expects to present a recommendation for a specification for heavy petroleum spirits.

The subcommittee recommends that the Tentative Methods of Sampling and Testing Turpentine (D 233 - 44 T) be continued as tentative without revision for another year.

Subcommittee VI on Definitions (Carlton H. Rose, chairman) recommends the following revisions for immediate adoption in the Standard Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 46):[2]

Bulking Value.—Change to read as follows:

> *Bulking Value.*—Solid volume of a unit weight of material usually expressed as gallons per pound. For practical purposes this is 0.120 divided by the specific gravity.

Tinting Strength.—Change to read as follows:

> *Tinting Strength.*—The power of coloring a standard paint or pigment.

The subcommittee also recommends the following new definitions for publication as tentative, to be added, when adopted, to standard D 16:

> *Plasticizer.*—A substance added to paint varnish or lacquer to impart flexibility.
>
> *Dope.*—A composition, usually a cellulosic lacquer, for application on textiles and leathers.
>
> *Mass Color.*—The color when viewed by reflected light of a pigment-vehicle mixture of such thickness as to obscure completely the background. Sometimes called over-tone or mass-tone.
>
> *Over-Tone.*—See Mass Color.
>
> *Mass-Tone.*—See Mass Color.
>
> *Undertone.*—The color of a thin layer of pigment vehicle mixture applied on a white background.

Subcommittee VII on Accelerated Tests (H. A. Nelson, chairman) is actively engaged in preparing methods of test covering humidity testing, immersion testing, and the preparation of steel panels.

The subcommittee is also conducting cooperative work on accelerated testing with the Cleveland Paint and Varnish Production Club.

Prepared discussion had been presented at the meeting of the subcommittee on subjects pertaining to (*1*) the operation of the light and water exposure apparatus which included comments on the use of deionized water, (*2*) the Bureau of Standards program on standardizing light sources, and (*3*) plans for general modifications of the light and water exposure apparatus as outlined by a manufacturer of such apparatus.

The subcommittee recommends that because of revisions under consideration, Tentative Specifications for Wood to be Used as Panels in Weathering Tests of Paint and Varnish (D 358 - 44 T) be continued as tentative.

The subcommittee also recommends that the Tentative Method of Evaluating Degree of Resistance to Flaking (Scaling) of Exterior Paints of the Linseed-Oil Type (D 772 – 44 T) be adopted as standard.

Subcommittee VIII on Methods of Chemical Analysis of Paint Materials (C. Y. Hopkins, chairman) recommends a revision for immediate adoption of Standard Methods of Testing Liquid Driers (D 564 – 43), consisting of changing the figure "15" to read "75" in Section 13 (*c*), line 16.

The subcommittee recommends that the tentative revision[2] of Standard Methods of Chemical Analysis of White Pigments (D 34 – 39) be adopted as standard.

The subcommittee also recommends that the Standard Method of Chemical Analysis of Dry Cuprous Oxide (D 283 – 39) be revised, as appended hereto,[5] and reverted to tentative.

The subcommittee recommends that the Standard Method of Analysis of Diatomaceous Silica Pigment (D 719 – 45) be revised, as appended hereto,[5] and reverted to tentative.

The subcommittee recommends that the Tentative Methods of Chemical Analysis of Zinc Yellow Pigment (D 444 – 46 T)[2] be revised as follows:

Section 2.—In line 12, delete the sentence beginning "The weighed portion . . ."

Section 3.—In line 3, change "110 C." to read "105 to 110 C."

New Section.—Add the following as a new Section 14 on "Insoluble in 10 per cent Acetic Acid."

14. *Matter Insoluble in Acetic Acid* (*1:9*). Weigh accurately about 10 g. of the sample (Note) and place in a 600-ml. beaker. Add 300 ml. of acetic acid (1:9). Heat the mixture to 80 C. and maintain at 80 ± 5 C., while stirring, until nothing further dissolves. Filter while hot through a tared Gooch crucible. Wash the insoluble residue on the filter with hot water. Dry the crucible at 105 to 110 C. and weigh. Matter insoluble in acetic acid (1:9) =

$$\frac{\text{wt. of residue}}{\text{wt. of sample}} \times 100$$

NOTE.—If the pigment contains an organic treating agent, this treating agent should first be removed by washing with ether or chloroform.

Subcommittee IX on Varnish (J. C. Weaver, chairman) recommends a revision for immediate adoption of Standard Methods of Testing Oleoresinous Varnishes (D 154 – 43) as appended hereto.[5] The principle changes are as follows:

(*1*) Color continues with potassium dichromate as primary standards, but there are included as secondary standards the Gardner (1933) solutions of cobalt and ferric chlorides whose 18 numbers are based on the potassium dichromate standards.

(*2*) Viscosity continues to be reportable in poises by any suitable method, but the Gardner-Holdt bubble tube method which is in wide use in the industry, is included in detail as an optional method.

(*3*) Nonvolatile matter determination is more carefully described.

(*4*) Elasticity tests by Run Kauri Reduction are further refined by adoption of the procedure in Federal Specification TT-P-141a.

(*5*) Alkali resistance test procedure is more carefully defined and a schedule for reporting results is included.

(*6*) The introduction of methods for specific gravity and reactivity.

The subcommittee recommends the withdrawal of the Standard Method of Test for Reactivity of Paint Liquids (D 479 – 40) since this method is incorporated in the revision of Method D 154.

The subcommittee recommends a revision of Tentative Method of Test for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions (D 563 – 45 T) which consists of changing the note under Scope to read as follows: "Note.—The method is not applicable for the determination of phthalic anhydride in alkyd resins containing other dibasic acids, such as, maleic or fumaric; or modifying agents, such as, urea, melamine and phenolic resins."

The subcommittee is developing methods of tests for drying time, for nonvolatility, and flexibility. The subcommittee is also considering starting work on the development of tests for reactivity with basic pigments, color of dried varnish films, alkali resistance, and viscosity. Work is also contemplated on specifications and methods of tests for driers.

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[5] These revised methods and specifications were accepted by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part II.

Subcommittee X on Optical Properties (M. Rea Paul, chairman) is working on nine projects, including studies on daylight reflectance, goniophotometry, preparation of panels for the determination of apparent luminous reflectance of traffic paints for night viewing, methods of color specification, preparation of panels for gloss determination, and method of test for absolute hiding power. The subcommitee is preparing a method of test for small color differences of paints.

This subcommittee recommends the adoption as standard of Tentative Method of Test for Daylight 45 Degree, 0 Degree Apparent Reflectance of Paint Finishes (D 771 – 44 T) with the following revisions:

Change the words "apparent reflectance" to read "directional reflectance" throughout the method.

Section 2.—Add the following note to this section:

NOTE.—The quantity here defined is sometimes loosely called "diffuse reflectance," or simply "reflectance." Sometimes it is erroneously called "brightness," a term having a wholly different photometric meaning.

The subcommittee also recommends that in view of the revisions which are being considered that the Tentative Method of Test for Specular Gloss of Paint Finishes (D 523 – 44 T) be continued as tentative.[6]

The subcommittee is referring to Subcommittee VI on Definitions three proposed definitions for their consideration. These cover total reflectance, directional reflectance, and gloss.

Subcommittee XI on Resins (J. J. Mattiello, chairman) has been organized into two general sections, on methods and on materials. The section on methods will consider measurement of nonvolatile content, acid value, saponification value, softening point, viscosity, refractive index, and qualitative analysis. The section on materials will consider the various types of resins.

Subcommittee XIII on Shellac (C. C. Hartman, chairman) is reviewing the specifications under its jurisdiction and also specifications on shellac of other specification writing organizations. The subcommittee expects to recommend changes in the shellac specifications within the coming year. The subcommittee, therefore, recommended that Tentative Specifications for Orange Shellac and other Lacs (D 237 – 43 T) and Tentative Method of Test for Color of Orange Shellac (D 29 – 41 T) be continued as tentative.

Subcommittee XV on Pigments (C. L. Crockett, chairman) recommends for publication as tentative Specifications for Mercuric Oxide for Use in Anti-Fouling Paints,[4] and Specifications for Dry Cuprous Oxide for Use in Anti-Fouling Paints.[4]

In view of these proposed tentatives the subcommittee recommends the withdrawal of Standard Specifications for Toxic Ingredients in Anti-Fouling Paints (Dry Cuprous Oxide, Dry Mercuric Oxide) (D 277 – 31).

The subcommittee has studied the question of uniformity of specifications covering dry pigment and paste-in-oil with particular reference to the maximum and minimum percentages of pigment and oil content and the subcommittee recommended that editorial changes be made in all the pigment and paste and oil specifications under the jurisdiction of Committee D-1 as follows:

Requirement, per cent	Change in Figures
Pigment	Round off to unity
Volatile matter	Round off to one decimal
Any other minor ingredients	Round off to one decimal
Skins and coarse particles	Round off to one decimal

In addition, the subcommittee recommends that only a minimum requirement for pigment be employed and that the per cent oil or nonvolatile vehicle be shown as "remainder."

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[6] A report prepared by R. S. Hunter discusses the suggested revisions of Method D 523 and has appended the completed suggested revised method. This report appears in the ASTM BULLETIN, No. 150, January, 1948, p. 72.

The subcommittee recommends for immediate adoption a revision of Standard Specifications for Iron Blue (D 261 – 46) to consist of: (*1*) In Section 2(*a*) change the second sentence to read "..., in small amounts up to a total of 5 per cent, ... " and in the same section change the maximum allowable moisture and other volatile matter from "7.0 per cent" to read "8.0 per cent", and (*2*) add the following paragraph to Section 4:

> (*b*) *Moisture.*—Mix 50 g. of the sample in a 250-ml. flask with 150 ml. of toluene. Place the flask in an oil bath, connect with a condenser, apply heat to the bath, and distill until about 100 ml. of distillate have been collected in a graduate. The number of milliliters of water collected under the toluene in the receiver, multiplied by two, is the percentage of water in the pigment.

The subcommittee also recommends for immediate adoption a revision of the Standard Specifications for Zinc Dust (Metallic Zinc Powder) (D 520 – 41) as appended hereto.[5]

The subcommittee recommends that the specifications under the jurisdiction of Committee D-1 for paste colors be revised by deleting the paragraph covering paste in Japan. This change would be an immediate revision of the following nine Standards: D 209 – 46, D 210 – 46, D 262 – 46, D 85 – 41, D 261 – 46, D 212 – 46, D 213 – 46, D 211 – 43, and D 264 – 41. The subcommittee also recommends that in these nine standards the sections on "Methods of Testing" be editorially revised to list the A.S.T.M. methods applicable in each case.

The subcommittee recommended that the following specifications be continued as tentative:

Tentative Specifications for:

Raw Umber (D 763 – 44 T),
Burnt Umber (D 764 – 44 T),
Raw Sienna (D 765 – 44 T),
Burnt Sienna (D 766 – 44 T), and
Venetian Red (D 767 – 44 T).

The subcommittee recommends that the tentative revision[2] of the Standard Specifications for C.P. Zinc Yellow (Zinc Chromate) (D 478 – 41) be adopted as standard. The subcommittee also recommends that Tentative Specifications for Yellow Iron Oxide, Hydrated (D 768 – 46 T) be adopted as standard.

The subcommittee is engaged in writing three new specifications which are not yet completed: for calcium carbonate, copper phthalocyanene blue, and copper powder for anti-fouling paint, and is also reviewing specifications for aluminum pigments, red pigments, titanium dioxide, and glazing putty.

Subcommittee XVIII on Physical Properties of Materials (R. H. Sawyer, chairman) is preparing a method of test for resistance to sand abrasion and is also considering a revision of the standard method for measuring oil absorption of pigments and specific gravity.

The subcommittee has work in progress on the measurement of the adhesion of paint films and the chalk resistance of titanium pigments.

The subcommittee recommends that the tentative revision[2] of the Standard Method of Test for Consistency of Exterior House Paints and Enamel-Type Paints (D 562–44) be adopted as standard.

The subcommittee also recommends an extensive revision of the Tentative Methods for Producing Films of Uniform Thickness of Paint, Varnish, Lacquer and Related Products on Test Panels (D 823 – 45 T), as appended hereto.[5]

Subcommittee XXV on Cellulosic Coatings and Related Products (R. M. Carter, chairman) recommends for publication the new proposed Tentative Methods of Testing Ethylcellulose.[4]

The subcommittee also recommended

that Tentative Method of Test for Ester Value of Tricresyl Phosphate (D 268-46 T) and Tentative Methods of Testing Cellulose Acetate (D 871 - 46 T) be continued as tentative.

Subcommittee XXIX on Painting of Structural Iron and Steel (A. J. Eickhoff, chairman) recommends that a report on "Study of the Pretreating and Painting of Steel" be approved for inclusion in the 1947 *Proceedings* of the Society.

This report has been submitted to letter ballot of the committee, which consists of 298 voting members; 133 members returned their ballots, of whom 111 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

W. T. PEARCE,
Chairman.

C. H. ROSE,
Secretary.

APPENDIX

REPORT ON THE EVALUATION OF SURFACE TREATMENT OF STEEL PRIOR TO PAINTING

Prepared by Arnold J. Eickhoff[1] on Behalf of Subcommittee XXIX on Painting of Structural Iron and Steel of Committee D-1

This is a final report of outdoor exposure tests conducted by Subcommittee XXIX of A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products to determine the effect of cold phosphate-phosphoric acid pretreatments of steel surfaces which were subsequently painted. A progress report[2] was published in 1941.

Pretreatments

The pretreatments investigated were essentially a mixture of water, phosphoric acid, chromates, reduced chromates, wetting agents, and metallic phosphates. The hot type phosphate–phosphoric acid treatments, such as those used extensively on automobile bodies and refrigerators, have proved advantageous for increasing paint adhesion and for retarding the spread of corrosion between the paint film and the steel surface. However, these hot types are not practical for application to structural steel already erected in the field. Hence the question, "How satisfactory are the cold types?"

Paint formulation was not considered in this project. A considerable amount of such work has been reported previously, for example, the Havre de Grace Bridge Tests[3] some forty-one years ago; reports on Atlantic City Steel Plate Tests[4] some thirty-six years ago; and the reports of Subcommittee XIV[5] about twenty-six years ago.

The cold washes used in this investigation, together with a general description and directions for their use, as supplied by their respective manufacturers, are as follows:

Pretreatment X was a mixture of chromic acid, phosphoric acid, a wetting agent, and some zinc dissolved in the phosphoric acid. The temperature of application was 80 F., the drying time was one hour. The steel surface should not be rinsed prior to application of the paint when this pretreatment is used.

Pretreatment Y was a saturated solution of zinc phosphate in an aqueous solution of phosphoric acid. The temperature of the solution when applied was 80 F., and the drying time was four hours. The work was then rinsed with water

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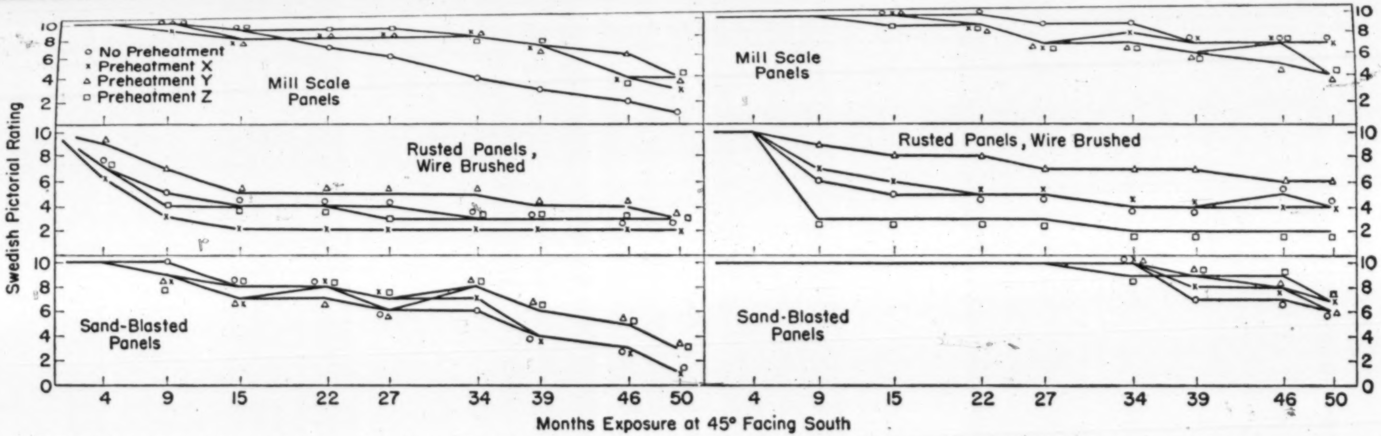
[1] National Lead Co., Research Laboratories, Brooklyn, N. Y.; chairman of Subcommittee XXIX.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 322 (1941).

[3] Report of Committee E on Preservative Coatings for Iron and Steel, *Proceedings*, Am. Soc. Testing Mats., Vol. VI, p. 47 (1906).

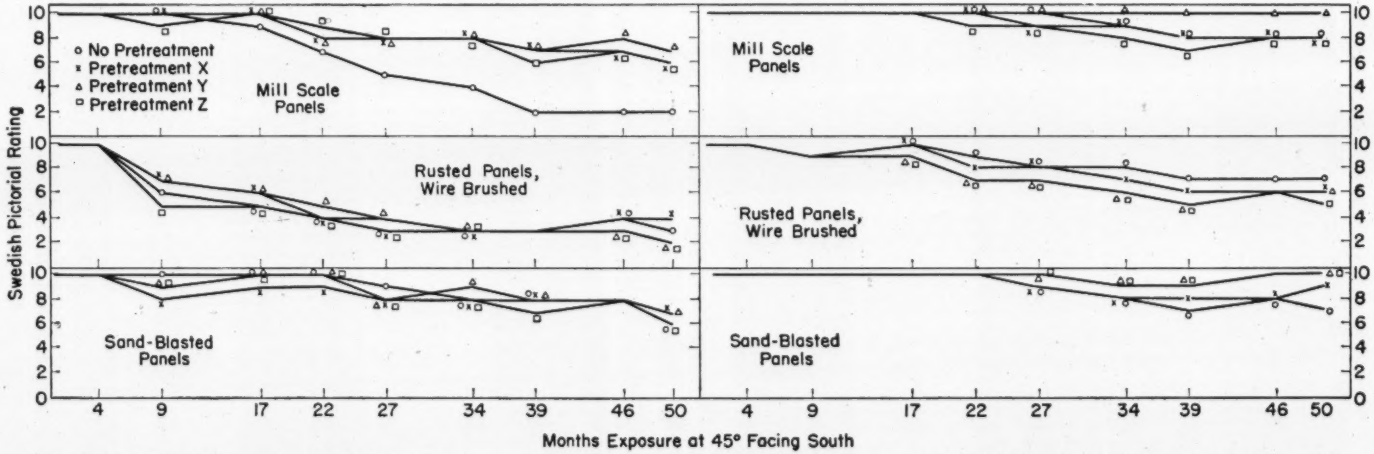
[4] Report of Subcommittee D of Committee D-1 on Preservative Coatings for Structural Materials, *Ibid.*, Vol. XI, p. 192 (1911).

[5] Report of Subcommittee XIV on the Preservation of Iron and Steel Surfaces for Painting of Committee D-1 on Preservative Coatings for Structural Materials, *Ibid.*, Vol. XIX, Part I, p. 374 (1919).



(a) Paint A on Panels Exposed at Sayville, Long Island, N. Y.

(b) Paint B on Panels Exposed at Sayville, Long Island, N. Y.



(c) Paint A on Panels Exposed at Bethlehem, Pa.

(d) Paint B on Panels Exposed at Bethlehem, Pa.

Fig. 1.—Swedish Pictorial Ratings of Panels Exposed at Bethlehem, Pa., and Sayville, Long Island, N. Y.

TABLE I.—MONTHS TO REACH SWEDISH RATINGS OF 8 AND 6 AND FINAL SWEDISH AND A.S.T.M. RATINGS OF ALL PANELS

Panel	Paint	Original Surface Condition[a]	Sayville, L. I., N. Y.						Bethlehem, Pa.				
			Pre-treatment	Spreading Rate of Primer	Months to Reach Swedish Rating of:		Final Rating at 50 Months		Spreading Rate of Primer	Months to Reach Swedish Rating of:		Final Rating at 50 Months	
					8	6	Swedish	A.S.T.M.		8	6	Swedish	A.S.T.M.
No. 1........	A	M.S.	None	962	19	27	1	> 4-2	800	19	24	2	> 4-1
No. 2........	B	M.S.	None	1015	38	50+	7	5-2	775	39	50+	8	8-2
No. 3........	A	R. & W.B.	None	934	3	9	3	> 4-2	800	6	9	3	> 4-1
No. 4........	B	R. & W.B.	None	831	7	9	4	4-2	650	27	50+	7	8-1
No. 5........	A	S.B.	None	883	15	27	1	> 4-1	800	34	50	6	7-1
No. 6........	B	S.B.	None	871	37	50	6	4-1	775	34	50+	7	8-1
No. 7........	A	M.S.	X	815	15	42	3	> 4-2	800	22	50	6	7-1
No. 8........	B	M.S.	X	1015	25	50+	7	5-2	790	39	50+	8	9-2
No. 9........	A	R. & W.B.	X	757	2	9	2	> 4-2	650	8	17	4	4-1
No. 10.......	B	R. & W.B.	X	926	8	15	4	4-2	775	22	39	6	7-1
No. 11.......	A	S.B.	X	935	12	36	1	> 4-1	695	9	50+	7	7-1
No. 12.......	B	S.B.	X	795	39	50+	7	8-1	775	34	50+	9	9-1
No. 13.......	A	M.S.	Y	722	15	46	4	4-2	820	22	50+	7	8-1
No. 14.......	B	M.S.	Y	746	25	39	4	4-2	775	50+	50+	10	10
No. 15.......	A	R. & W.B.	Y	739	8	12	3	> 4-2	800	8	17	2	> 4-1
No. 16.......	B	R. & W.B.	Y	732	15	46	6	5-2	675	19	34	6	6-1
No. 17.......	A	S.B.	Y	756	12	27	3	4-1	710	27	50+	7	8-1
No. 18.......	B	S.B.	Y	831	46	50	6	6-1	630	50+	50+	10	10
No. 19.......	A	M.S.	Z	962	34	42	4	> 4-2	820	27	39	6	7-1
No. 20.......	B	M.S.	Z	914	25	39	4	4-2	775	34	50+	8	8-2
No. 21.......	A	R. & W.B.	Z	883	3	6	3	> 4-2	800	6	8	2	> 4-1
No. 22.......	B	R. & W.B.	Z	892	6	7	2	> 4-2	725	19	34	5	7-1
No. 23.......	A	S.B.	Z	794	15	40	3	> 4-1	780	27	50	6	7-1
No. 24.......	B	S.B.	Z	892	48	50+	7	7-1	725	50+	50+	10	10

[a] M.S. indicates Mill Scale, R. & W.B. indicates Rusted and Wire Brushed, S.B. indicates Sandblasted.

and dried for one hour prior to application of the paints.

Pretreatment Z was a homogeneous mixture of phosphoric acid, chromic acid phosphate, water-soluble oil solvent, a wetting agent, and water. The temperature of the solution when applied was approximately 130 F. This solution was applied by brush in sufficient quantity merely to wet the surface. The panels were dried two hours and the grayish deposit formed from the chemical pretreatment was allowed to remain.

Test Surfaces and Paints

In this study the panels used were 2 by 3 ft. by $\frac{3}{16}$-in. hot-rolled steel, with a copper content of not more than 0.06 per cent. In choosing such a large panel, it was believed the results of the tests would be more indicative of actual practice.

Three surface conditions were chosen: one was the original hot-rolled panel with mill scale—the grease and dirt removed with volatile petroleum solvent. The second was the mill-scale panel, considerably rusted, then wire brushed before any pretreatment or paint was applied. The third was a mill-scale panel, sandblasted prior to the application of paint or pretreatment.

Two exposure locations were selected—Bethlehem, Pa. (an industrial atmosphere), and Sayville, Long Island, N. Y. (a rural atmosphere). In addition to the control panels which were not given any pretreatment, each of the surfaces was separately treated with three washes of the phosphate type, before the paint was applied.

There were only two paint systems used, designated system A and system B—each system comprising a primer and a topcoat. The paints used in system A were of inferior quality and purposely selected to produce a coating which would not provide prolonged protection. The paints used in system B were considered to be of better quality so that long-time protection would be provided. A total of 24 panels was exposed at each location. The angle of exposure was 45 deg. facing south.

Pictorial Corrosion Standards

Since the committee was chiefly interested in corrosion as an index of protection, a numerical corrosion rating system was desirable. Several sets of Swedish pictorial rusting standards were obtained and, as these were the only pictorial standards then available, they were adopted as a basis for rating corrosion. At a later date the A.S.T.M. Standard Method for Evaluating Degrees of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces (D 610 – 43)[6] was developed.

In Fig. 1 are shown the progressive ratings for the 50-month test period for each of the paints at each of the locations on panels which had received no pretreatment and on panels given pretreatments *X*, *Y*, and *Z*, respectively. In part (*a*) of this figure is shown the record of paint A on panels exposed at Bethlehem, Pa., and part (*b*), the record of paint B at the same location; part (*c*) is the record of paint A on panels exposed at Sayville, L. I.;

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[6] 1946 Book of A.S.T.M. Standards, Part II, p. 1186.

TABLE II.—RATIO (IN PER CENT) OF MONTHS TO REACH SWEDISH RATINGS OF 8 AND 6 FOR PRETREATED *VERSUS* UNTREATED SHEETS.

(Three types of original surfaces.)

Swedish Rating	Pre-treatment	Mill Scale				Wire Brushed				Sandblasted			
		Sayville		Bethlehem		Sayville		Bethlehem		Sayville		Bethlehem	
		Paint A	Paint B	Paint A	Paint B	Paint A	Paint B	Paint A	Paint B	Paint A	Paint B	Paint A	Paint B
8	X	79	66	116	100	67	114	133	81	80	105	26	100
8	Y	79	66	116	128	267	214	133	70	80	124	79	147+
8	Z	179	66	142	87	100	86	100	70	100	130	79	147+
6	X	156	100±	208	100±	100	167	189	78−	133	100+	100+	100±
6	Y	170	78−	208+	100±	133	511	189	68−	100	100	100+	100±
6	Z	156	78−	163	100±	67	78	89	68−	148	100+	100	100±

and (*d*) the record of paint B at the same point.

Some of the same data are given in Table I which shows for the two paint systems and the several treatments and original surface conditions, the months required to reach ratings of 8 and 6 on the Swedish scale at the two locations. The table also affords a comparison of the Swedish and A.S.T.M. ratings at the conclusion of the tests after 50-month exposure.

The Swedish pictorial rating of corrosion ranges from 0 to 10. A rating of 10 indicates no failure, zero indicates complete failure. These ratings are given for the three different surfaces, and the three pretreatments.

The A.S.T.M. method for evaluating degrees of rusting is also a numerical scale. Number 10 is a surface with practically no blemishes, while the lower numbers indicate progressively worse conditions. However, No. 4 is the worst condition classified under the A.S.T.M. system and the pictorial standards are further divided into two types. Type 1 indicates that the rusting is not accompanied by blistering, while type 2 indicates that the rusting is accompanied by blistering (in the A.S.T.M. system 6-2 indicates a type 2 rusting with a rating of 6).

It will be noted from Table I that in classifying the end-point evaluation of the panels there is, for the most part, reasonably good numerical agreement between the Swedish and the A.S.T.M. systems. Occasionally, one finds a difference, chiefly due to the fact that the A.S.T.M. numbers do not go down so low as the Swedish. On the whole, it is surprising that two pictorial systems developed on the opposite sides of the Atlantic Ocean have such relatively good agreement.

DISCUSSION OF DATA

Data on the number of months required to reach the Swedish ratings of 8 and 6 are given in Table I. At a rating of 8, repainting is generally considered economically desirable. At a rating of 6, repainting is generally considered to be imperative and extensive surface preparation is required if the paint is to afford reasonable future protection. It will be noticed that at the end of 50 months there are 16 panels which did not reach a corrosion rating of 6. Therefore, the information obtained or the conclusions which can be drawn are somewhat limited.

From the data given in Table I, Table II has been prepared to show the ratio (in per cent) of the number of months required to reach Swedish ratings of 8 and 6, respectively, for pretreated panels *versus* untreated sheets. The committee concluded that small differences in these percentage ratings were not too significant and arbitrarily grouped them before conclusions were drawn.

A percentage between 85 and 115 indicated no advantage or disadvantage for the pretreatment; for one between 85 and 65 per cent the pretreatment was considered disadvantageous; 65 per cent or less the pretreatment was definitely disadvantageous; between 115 to 135 per cent the pretreatment was considered slightly advantageous; at 135 per cent or more the pretreatment was considered definitely advantageous. On the basis of these percentage limits, the following conclusions are noted.

CONCLUSIONS

From the data obtained it is readily noted that there is no consistent advantage or disadvantage in the indiscriminate use of the pretreatments on steel exposed to the atmosphere.

It should be noted that if the mill scale is tightly adherent over the entire surface, a condition seldom obtained from a practical standpoint, the paint performance is not adversely affected. This would explain the equally excellent performance of the mill-scale surfaces compared with the sandblasted surfaces.

Wire brushed, rusted panels gave very short paint life under all conditions.

FUTURE STUDY

Most metal protective paint studies which have been reported in the technical literature have concerned themselves with the preparation and painting of new, unweathered steel, or steel cleaned by various mechanical means. Our subcommittee decided that valuable information would be derived from a study of the effect of cleaning, pretreating, and repainting steel surfaces which had received no pretreatment and surfaces which had received mechanical and chemical pretreatments. Consequently, the second phase of this work is concerned with the preparation and painting of the same original painted and weathered panels reported earlier in this report. It is of practical interest to know whether the repainted panels will perform in the same manner as when originally painted. This second phase of the original investigation is now under way and the panels are exposed at the same locations—Sayville, Long Island, N. Y., and Bethlehem, Pa.

APPENDIX

In order that there be no misunderstanding, and to avoid giving the impression that these cold phosphate washes are of questionable value, attention is called to the work done by the U. S. Army Engineers and the Tennessee Valley Authority. Invariably, these cold pretreatments proved of value when used on sandblasted steel surfaces subsequently painted and submerged in fresh water.

Under these conditions of exposure, chemical pretreatment extended the protective life of the applied paint coatings. On the other hand, the use of these same pretreatments applied in a similar manner did not significantly extend the protective life of the paint coatings exposed to atmospheric environments, which finding was developed as the result of the work of Subcommittee XXIX.

REPORT OF COMMITTEE D-2*

ON

PETROLEUM PRODUCTS AND LUBRICANTS

Committee D-2 on Petroleum Products and Lubricants held three regular meetings in 1947. The January and March meetings were held in Washington, D. C. The third was held in Atlantic City, N. J., in June.

CHANGES IN COMMITTEE ORGANIZATION

During the year ending June 18, 1947, the following changes in the organization of the technical committees and subcommittees have been made or authorized:

Technical Committee A on Gasoline has been reorganized; Section II on Corrosion, C. F. Ramey, chairman, and Section IV on Detonation, D. B. Brooks, chairman, have been discharged. Section V on Specifications is being reorganized under the chairmanship of H. C. Dickinson.

Technical Committee F on Diesel Fuels has been organized and held its first meeting in the form of a "Forum on Diesel Fuels" in Chicago, Ill., on November 13, 1946. Papers presented at the Forum have been published by the Society as Technical Publication No. 71.[1] Officers of the technical committee are, C. G. A. Rosen, chairman; J. R. Jackson, vice-chairman; and W. G. Ainsley, secretary. Sections I for Liaison with CRC Committees, W. J. Backoff, chairman; and II on Specifications, H. M. Gadebusch, chairman, have been organized.

Technical Committee H on Light Hydrocarbons, W. G. Lovell, chairman; R. C. Alden, secretary, has completed its organization and gone to work. Sections I on Screening, R. C. Alden, chairman; II on Natural Gasoline, R. C. Alden, chairman; III on Chemical Utilization Including Pure Hydrocarbons, R. F. Dunbrook, chairman; IV on Testing Methods, B. R. Carney, chairman; V Editorial and Nomenclature, F. D. Tuemmler, chairman; and VI on Liquefied Petroleum Gas, G. R. Benz, chairman, have been organized. The activities of Section VI have been correlated with Committee D-3 on Gaseous Fuels.

Technical Committee J on Aviation Fuels has completed its organization and is at work. Officers are G. K. Brower, chairman, and Albert E. Miller, secretary. Six sections have been organized, or are being organized; I on Specifications, J. T. Hendren, chairman; II on Detonation; III on Stability; IV on Volatility, O. C. Bridgeman, chairman; V on TEL Content, R. V. Kerley, chairman, and VI on Fuel Deposits in Power Plants.

Technical Committee K on Cutting Oils held its first meeting on March 21, 1947, in the form of a Symposium on Cutting Oils. Officers of the committee are, O. L. Maag, chairman, E. A. Reehl, vice-chairman, and W. J. Backoff, secretary.

Section A on Vacuum Distillation of Subcommittee VIII on Distillation has been organized under the chairmanship of H. M. Smith.

Section D on Units of Measurement Calculation and Tables, B. J. Heinrich, chairman, of Subcommittee XV on Measurement and Sampling, has been reorganized and enlarged. This section is developing reference tables for volume correction of petroleum products which may, if successful, become internationally recognized standards.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Forum on Diesel Fuel Oils, Am. Soc. Testing Mats., (March, 1947). Issued as a separate publication.

A Division on Cumbustion Characteristics has been organized with seven sections. This step was taken in recognition of the responsibilities entailed in the standardization of methods of test for knock rating of engine fuels. The organization of the division is reflected in the following list:

Division on Combustion Characteristics:
Donald B. Brooks, Chairman,
A. E. Becker, Vice-Chairman,
H. M. Trimble, Secretary.
Sections:
I on Exchange Testing, Gustav Wade, chairman,
II on Precision of Methods, J. M. Campbell, chairman,
III on Instrumentation, J. E. Taylor, chairman,
IV on Test Equipment, B. R. Siegel, chairman,
V on Reference Materials, F. C. Burk, chairman,
VI on Operation and Maintenance, C. E. Haberman, chairman, and
VII, Editorial, A. E. Becker, chairman.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1946 Annual Meeting, Committee D-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:
Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils (D 893 - 46 T), and
Test for Sulfur in Lubricating Oils Containing Additives and in Additive Concentrates by Bomb Method (D 894 - 46 T), and
Test for Foaming Characteristics of Crankcase Oils (D 892 - 46 T).

The first two recommendations were accepted by the Standards Committee on November 6, 1946, and the third on December 5, 1946. The new tentatives appear in the 1946 Book of A.S.T.M Standards, Part III-A, and the 1946 Compilation of A.S.T.M. Standards on Petroleum Products and Lubricants.

ACTIVITIES OF TECHNICAL COMMITTEES AND SUBCOMMITTEES

Technical Committee A on Gasoline (B. B. Bachman, chairman).—In recognition of the fact that the Research Method for determination of knock rating of motor fuels and the Supercharge Method for knock characteristics of aviation gasoline have become important standards in commerce, this committee has proposed that these methods be published as tentative, as appended here to.[2]

Technical Committee B on Lubricating Oils (W. S. James, chairman) held an informal symposium in June, 1947, on the activities of Technical Committee B and its relationship to the activities of other organizations outside A.S.T.M. which work in the field of lubricating oils.

Technical Committee C on Turbine Oils (F. C. Linn, chairman) developed the Proposed Recommended Practices for Preparation of New Turbine Lubricating Systems which appear in Appendix I.[3] The Technical Committee has recommended that this also be published in technical and trade journals used by the operators of steam turbines. A Method of Test for Oxidation Characteristics of Inhibited Steam Turbine Oils has been developed and is offered for publication as tentative.[2] In addition, it is proposed that Method D 665 - 46 T, Test for Rust-Preventing Characteristics of Steam Turbine Oils in the Presence of

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[2] See Editorial Note, p. 279.
[3] See p. 289.

Water be revised to include a test with synthetic sea water.

Technical Committee G on Lubricating Grease (F. L. Wright, chairman) has made the following three proposals: (*1*) Revision of Method D 217, Test for Cone Penetration of Lubricating Grease[2] (*2*) revision of Method D 128, Grease Analysis,[4] and (*3*) a new Tentative Method of Test for Oxidation Stability of Lubricating Greases by the Oxygen Bomb Method.[2] Technical Committee G, in connection with its studies of evaporation of lubricating grease, has investigated and modified a Method of Test for Evaporation Loss of Lubricating Greases and Oils developed by the Coordinating Lubrication Research Commitee of the Coordinating Research Council, Inc. The modified test is published as information in Appendix II.[5]

Technical Committee H on Light Hydrocarbons (W. G. Lovell, chairman) has been investigating methods for determination of butadiene in hydrocarbon mixtures. The Koppers-Hinckley Method of Test for Determination of Butadiene Content of Polymerization Grade Butadiene appears promising and is published as information in Appendix III.[6]

The results of the Cooperative Test Program conducted by the Office of Rubber Reserve on Reproducibility and Repeatability of this method are presented in a special report.[7] This technical committee has under way the development of a precision method for determination of vapor pressure of petroleum products.

Technical Committee J on Aviation Fuels (G. K. Brower, chairman) prepared the new Tentative Specifications for Aviation Gasoline.[2] In addition, Technical Committee J, along with Technical Committee A on Gasoline, proposed that the Method of Test for Knock Characteristics of Aviation Fuels by the Supercharge Method, after revision by the Division on Combustion Characteristics be published as tentative.

Subcommittee I on Pharmaceutical Tests (G. M. McNulty, chairman) and *Subcommittee III on Paraffin Wax* (J. B. Rather, Jr., chairman) have rewritten part II of Method D 217 - 44 T as a separate procedure for the determination of penetration of petrolatum. The necessity for this development arose from the fact that Technical Committee G on Lubricating Grease has proposed a revision of Method D 217 - 44 T, Test for Consistency of Lubricating Grease and Petrolatum, which leaves out the part of Method D 217 applicable to petrolatum. The rewritten procedure, which incorporates certain changes suggested by the Subcommittee on Coordination of Test Methods, is proposed as tentative.[2]

Subcommittees I and III have engaged in a joint program of investigation of congealing points of pharmaceutical petrolatums. A new Tentative Method of Test for Congealing Point of Pharmaceutical Petrolatums[2] has been developed. This test is an alternate to Method D 127 - 30, Test for Melting Point of Petrolatums.

Subcommittee III has carried out a program to improve the reproducibility of Method D 721 - 44, Test for Oil Content of Paraffin Wax, and has developed in cooperation with Committee E-I on Methods of Testing, a new armoured thermometer for use with the method. This subcommitee has proposed extensive editorial revision of Method D 721 to accomplish the desired purpose.[4]

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[4] These revised methods and definitions were accepted by the Society and appear in the 1947 Supplement to Book of A.S T.M. Standards, Part III-A.

[5] See p. 297.

[6] See p. 300.

[7] See p. 283.

Subcommittee V on Viscosity (J. C. Geniesse, chairman) held an informal symposium on viscosity measurement at high rates of shear at its meeting at Atlantic City, on June 16, 1947.

At the June meeting, this subcommittee recommended publication of a new viscosity temperature chart E covering temperatures from −100 F. to +450 F. and a kinematic viscosity range of 2.0 to 20,000,000 centistokes. Suitable editorial changes in D 341 were recommended to include references to this new chart which covers the same viscosity range as chart C but extends the lowest temperature from −30 F. to −100 F.

Subcommittee VI on Color (H. M. Hancock, chairman) has been studying photoelectric methods of color determination. A method under consideration appears as information in Appendix IV.[8] The subcommittee proposes continuation of Method D 155 Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter as tentative without change because the method is generally regarded as unfit for adoption as standard, and no basic improvements can be suggested at this time. The subcommittee also proposed continuation of the Emergency Method ES-32, Color of U.S. Army Motor Fuel (All Purpose) by Means of an A.S.T.M. Color Standard because of a request from Army Ordnance.

Subcommittee VII on Sulfur (H. M. Hancock, chairman) has engaged in a study to improve the accuracy of the lamp sulfur determination. A new CO_2-O_2 Method for Determination of Sulfur Content of Petroleum Products by Lamp Method is published in Appendix V[9] as information. Because of the inaccuracies of the present Lamp Sulfur Method D 90, the subcommittee has proposed a revision[2] of this tentative method in which a gravimetric procedure is substituted for the titrimetric procedure used heretofore.

Subcommittee X on Insoluble Content of Used Oils (L. L. Davis, chairman) has continued to study methods for evaluation of used lubricating oil. A research procedure for determination of the mineral oil content of used lubricating oil has been developed and is published as information in Appendix VI[10]. In addition, the subcommittee has proposed that Method D 893 - 46 T, Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils, be continued as tentative without change.

Subcommittee XI on Inorganic Elements in Lubricants (C. M. Gambrill, chairman) has submitted a report[11] in support of the proposals for revision of Method D 874 – 46 T, Test for Sulfated Residue from New Lubricating Oils and Method D 810 – 44 T, Chemical Analysis for Lead, Copper, and Iron in Lubricating Oils.

Subcommittee XIII on Neutralization Number and Saponification (H. P. Ferguson, chairman) has under consideration the two methods for determination of acid and base numbers using color-indicators, published in Appendices VII[12] and VIII[13] as information. In addition, Subcommittee XIII proposed that the Method of Test for Saponification Number of Petroleum Products by Electrometric Titration, published as information in Appendix I to the 1943 Report of Committee D-2,[14] be published as tentative.

Subcommittee XVI on Cloud and Pour Points (L. C. Beard, Jr., chairman) has made an extensive review of the work done during the past few years covering laboratory methods and field storage tests. The subcommittee has reported that its studies have shown that the proposed Method for Determination of Maximum Pour Points of Lubricating Oils Containing Pour Point Depressants, published as information in Appendix I to the 1945 Report of Committee D-2[15] was found to give satisfactory results (within 10 F. of that found during field storage) in only 50 to 60 per cent of the samples tested. To date, the results do not promise the development of a short-time pour test. The subcommittee has proposed a revision of Method D 97 - 39, Test for Cloud and Pour Points, to allow use of the A.S.T.M. Low Cloud and Pour Thermometer with range −112 to 70 F., which was manufactured during the war, for use at low temperatures under the former EA-D 97.

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[8] See p. 307.
[9] See p. 315.
[10] See p. 322.
[11] See p. 280.
[12] See p. 327.
[13] See p. 331.
[14] *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 265 (1943).

Subcommittee XVIII on Aniline Point (Eugene Ayres, chairman) has recommended an extensive revision[2] of Method D 611 - 46 T, Test for Aniline Point and Mixed Aniline Point of Petroleum Products, to include products darker than No. 8 A.S.T.M. Color. The revision is necessary to get satisfactory accuracy and reproducibility.

Subcommittee XIX on Illuminating Oils (E. W. Dean, chairman) has undertaken to provide suitable alternates for certain items of unobtainable equipment, which are specified in Method D 187 - 39, Test for Burning Quality of Kerosine Oils. The conclusion has been that by using the apparatus specified in the Institute of Petroleum method, and adhering to the operating procedure now prescribed, the desired end can be attained. Accordingly, a suitable tentative revision of Method D 187 has been proposed.[2]

Subcommittee XX on Nomenclature, (Stewart S. Kurtz, Jr., chairman) has proposed some changes for Standard D 288 - 39, Definitions of Terms Relating to Petroleum, which are given later in this report.

Subcommittee XXV on Analysis of Petroleum Products for Hydrocarbon Types (Stewart S. Kurtz, Jr., chairman) has continued its extensive program of development of methods for analysis of petroleum products and has proposed the following three new methods as tentative: Test for Determination of Aromatics in Mixtures of Paraffins and Naphthenes, Test for Density by Pycnometer, and Test for Purity by Measurement of Freezing Points. In addition, this subcommittee has under consideration a Method of Test for Total Olefinic and Aromatic Hydrocarbons in Gasoline, published as information in Appendix IX.[16]

Division on Combustion Characteristics (Donald B. Brooks, chairman) has drafted the new Tentative Method of Test for Knock Characteristics of Aviation Fuels by the Supercharge Method[2] and Method of Test for Knock Characteristics of Motor Fuels by the Research Method.[2] These proposed tentative methods are revisions of the procedures developed by the Coordinating Fuel Research Committee of the Coordinating Research Council, Inc., and the revisions are in line with the proposals of Technical Committees A on Gasoline and J on Aviation Fuels. The Division has also, in expectation that the above methods will be recommended for tentative in accordance with the proposals of Technical Committees A and J, revised and brought up to date the following three methods: Knock Characteristics of Motor Fuels (D 357 - 46),[4] Knock Characteristics of Aviation Fuels (D 614 - 46 T),[2] and Ignition Quality of Diesel Fuels (D 613 - 43 T).[2]

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[15] *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 244 (1945).

[16] See p. 333.

The Division recommends that the five knock test methods be published in a Manual separate from the A.S.T.M. Standards on Petroleum Products and Lubricants, and that the manual include complete information on the operation and maintenance of knock test engines. Because of the volume of material involved, the supplementary information cannot be included in this report.

I. New Tentative Methods and Specifications

The committee recommends that the following ten methods and one specification be accepted for publication as tentative:

Tentative Methods of Test for:

Knock Characteristics of Motor Fuels by the Research Method,[2]
Knock Characteristics of Aviation Fuels by the Supercharge Method,[2]
Oxidation Stability of Lubricating Grease,[2]
Oxidation Characteristics of Inhibited Steam Turbine Oils,[2]
Penetration of Petrolatum,[2]
Congealing Point of Pharmaceutical Petrolatum,[2]
Saponification Number of Petroleum Products by Potentiometric Titration,[2]
Test for Aromatic Hydrocarbons in Mixtures with Naphthenes and Paraffins,[2] by Silica Gel Adsorption,[17]
Density by Pycnometer Method,[4]
Purity by Measurement of Freezing Points,[4] and

Tentative Specifications for:

Aviation Gasolines.[2]

II. Revision of Tentative Methods

The committee recommends revision of the following seven tentative methods as indicated:

Tentative Methods of:

Knock Characteristics of Aviation Fuels (D 614 - 46 T), revised as appended hereto.[2]
Ignition Quality of Diesel Fuels (D 613 - 43 T), revised as appended hereto,[2]
Consistency of Lubricating Greases and Petrolatum (D 217 - 44 T), revised as shown in the Proposed Tentative Method of Test for Penetration of Lubricating Grease,[2]
Sulfur in Petroleum Oils by Lamp Method (D 90 - 46 T), revised as shown in the Proposed Tentative Method of Test for Sulfur in Petroleum Products, Lamp Gravimetric Method,[2]
Sulfated Residue from New Lubricating Oils (D 874 - 46 T), revised by changing Section 1 on scope to read as follows:

1. *Scope.*—This method is intended for the determination of the quantity of sulfated residue obtainable from new lubricating oils and from lubricating oil additive concentrates. It may be used to indicate the concentration of known metal-containing additives in new oils. Sulfur, bromine, and chlorine do not interfere; phosphorus, when present in concentrations of 0.1 per cent or more, tends to interfere by remaining partially in the residue. This method is not applicable to new or used oils containing lead because of partial loss of lead; sulfated residue from such oils may be determined in accordance with A.S.T.M. Method D 810, Test for Sulfated Residue, Lead, Iron, and Copper in New and Used Lubricating Oils.

NOTE 1.—A.S.T.M. Method D 482, Test for Ash Content of Petroleum Oils, does not generally give comparable or related results when applied to additive-type oils and, in general, is not recommended for such oils.

Chemical Analysis for Lead, Copper, and Iron in Lubricating Oils (D 810 - 46 T), revised as shown in the Proposed Tentative Methods for Sulfated Residue, Iron, and Copper in New and Used Lubricating Oils,[2]
Aniline Point and Mixed Aniline Point of Petroleum Products (D 611 - 46 T), revised as appended hereto.[2]
Rust Preventing Characteristics of Steam Turbine Oil in the Presence of Water (D 665 - 46 T), revised as follows:

Section 5.—Change the heading of this section from "Procedure" to read "Procedure A for Distilled Water."

New Section.—Add the following new Section 6, relettering the subsequent section accordingly:

6. *Procedure B for Synthetic Sea Water.*—(*a*) The procedure for rust-preventing characteristics of steam turbine oils in the presence of synthetic sea water shall be identical with that described in Section 5 (*a*), (*b*), and (*c*), except that synthetic sea water shall be used in place of distilled water in that portion of the procedure described in Section 5 (*c*). The synthetic sea water shall have the following composition:

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[17] This Method was published in 1946 as information; see *Proceedings*, Am. Soc. Testing Mats., Vol. 46 (1946); also Compilation of A.S.T.M. Standards on Petroleum Products and Lubricants, October, 1946, p. 20.

Salt	grams per liter
$NaCl$	24.54
$MgCl_2 \cdot 6\ H_2O$	11.10
Na_2SO_4	4.09
$CaCl_2$	1.16
KCl	0.69
$NaHCO_3$	0.20
KBr	0.10
H_3BO_3	0.03
$SrCl_2 \cdot 6H_2O$	0.04
NaF	0.003

(*b*) The solution can be conveniently prepared as follows. The method avoids any precipitation in concentrated solutions with subsequent uncertainty of complete resolution. Using C. P. chemicals and distilled water, prepare the following stock solutions:

Stock Solution No. 1:

$MgCl_2 \cdot 6\ H_2O$	3885 g.
$CaCl_2$ (Anhydrous)	406 g.
$SrCl_2 \cdot 6\ H_2O$	14 g.

Dissolve and dilute to 7 liters.

Stock Solution No. 2:

KCl	483 g.
$NaHCO_3$	140 g.
KBr	70 g.
H_3BO_3	21 g.
NaF	2.1 g.

Dissolve and dilute to 7 liters.

To prepare the synthetic sea water, dissolve 245.4 g. of NaCl and 40.94 g. of Na_2SO_4 in a few liters of distilled water, add 200 ml. of Stock Solution No. 1 and 100 ml. of Stock Solution No. 2 and dilute to 10 liters. Stir the 10-liter batch and add 0.1 *N* Na_2CO_3 until the pH is between 7.8 and 8.2 One or two milliliters of the carbonate solution will be required.

III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in five standards, as set forth below, and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society.

Standard Methods of Test for:

Knock Characteristics of Motor Fuels (D 357 - 46), revised as appended hereto.[4]

Analysis of Grease (D 128 - 40), revised as appended hereto.[4]

Test for Oil Content of Paraffin Wax (D 721 - 44), revised as appended hereto.[4]

Test for Cloud and Pour Points (D 97 - 39), revised by changing Section 3 (*b*) to read as follows:

> (*b*) *Thermometer.*—An A.S.T.M. Cloud and Pour Test Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of −38 to +50 C. or −36 to +120 F.; or an A.S.T.M. Low Cloud and Pour Thermometer having a range of −80 to +20 C. or −112 to +70 F., and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1).

Also change footnote 3 to read as follows:

> [3] For tests above −65 F. it is permissible to use the A.S.T.M. Low Cloud and Pour Thermometers having ranges of −60 to +20 C. and −70 to +70 F., as previously prescribed for thermometers 6C-39 and 6F-39 in the Standard Specifications for A.S.T.M. Thermometers E 1 - 46, 1946 Book of A.S.T.M. Standards, Part III-A, p. 718.

Standard Definitions of Terms Relating to Petroleum (D 288 - 39), revised to include the following in proper alphabetical order:

> *Cymogene.*—This term is archaic and should not be used.
>
> *Rhigolene.*—This term is archaic and should not be used.

Delete from this standard the definition of End Point.

IV. REVISION OF STANDARD AND REVERSION TO TENTATIVE

In line with the proposal of Subcommittee XIX the committee recommends that the Standard Method of Test for Burning Quality of Kerosine Oils (D 187 - 39) be revised and that the revised method appended hereto,[2] be published as tentative.

V. EDITORIAL CHANGE IN STANDARD

It is recommended that the following note be inserted editorially in Section 1 of Standard Method D 217 - 30, Test for Melting Point of Petrolatum, provided that the new Tentative Method of

Test for Congealing Point of Pharmaceutical Petrolatum is accepted:

NOTE 1.—For testing pharmaceutical petrolatums, A.S.T.M. Method D 938 - 47 T, Test for Congealing Point of Pharmaceutical Petrolatums is recommended.

The reason for this recommendation is obvious.

VI. EMERGENCY STANDARD

The committee recommends that the Emergency Method of Test for Color of U. S. Army Motor Fuel (All Purpose) by Means of A.S.T.M. Color Standard (ES-32) be continued, as proposed by Subcommittee VI.

TABLE I.

Title	A.S.T.M. Designation	ASA Number
I. APPROVED AS AMERICAN STANDARD		
Test for Ash Content of Petroleum Oils	D 482-46	Z11.54—1947
II. REVISION OF AMERICAN STANDARDS		
Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products	D 86-46	Z11.10—1947
Test for Flash and Fire Points by Means of Open Cup....	D 92-46	Z11. 6—1947
Test for Flash Point by Means of the Pensky-Martens Closed Tester	D 93-46	Z11. 7—1947
Test for Water in Petroleum Products and Other Bituminous Materials ...	D 95-46	Z11. 9—1947
Test for Water and Sediment in Petroleum Products by Means of Centrifuge..................	D 96-46	Z11. 8—1947
Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue)	D 189-46	Z11.25—1947
Test for Knock Characteristics of Motor Fuels.	D 357 46	Z11.37—1947
Test for Gum Content of Gasoline................	D 381 46	Z11.36—1947

VII. TENTATIVES CONTINUED AS TENTATIVE

The committee recommends that the following methods and specifications which have been published as tentative for two years or longer without revision, be continued because revisions are under consideration or in prospect:

Tentative Method of Test for:

Color of Lubricating Oils and Petrolatum by Means of A.S.T.M. Union Colorimeter (D 155 - 45 T),
Chlorine in Lubricating Oils by Bomb Method (D 808 - 44 T),
Chemical Analysis for Phosphorus in Lubricating Oils (D 809 - 44 T),
Chemical Analysis for Metals in Lubricating Oils (D 811 - 44 T),
Normal Pentane and Benzene Insolubles in Used Lubricating Oils (D 893 - 46 T),

Tentative Specifications for:

Fuel Oils (D 396 - 39 T), and
Gasoline (D 439 - 40 T).

VIII. AMERICAN STANDARDS

Table I shows the changes in status for the year ending June 1, 1947, of American Standards for petroleum products, approved by the American Standards Association on the recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants, resulting from proposals made by Committee D-2. In addition, 32 A.S.T.M. Standards which have been American Standards for over three years without revision were reviewed and reaffirmed immediately as American Standards.

The committee proposes that the Society recommend to Sectional Committee Z11 that Method D 187 - 39 (ASA No. Z11. 17 - 1939), Test for Burning Quality of Kerosine Oils, be discontinued as American Standard.

The committee has no new standards to propose for American Standard this year.

The committee proposes that the Society recommend to Sectional Committee Z11 that the revisions to A.S.T.M. Standards listed in this report under Revisions of Standards, Immediate Adoption or such of these revisions as are approved by the Society, be approved immediately as American Standard.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 93 voting members, the results of which will be reported at the Annual Meeting.[18]

This report has been submitted to letter ballot of the committee which consists of 93 voting members; 71 members returned their ballots, of whom 62 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. DANTSIZEN,
Chairman.

W. T. GUNN,
Secretary.

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[18] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Knock Characteristics of Aviation Gasoline by the Supercharge Method (D 909 - 47 T), and
Knock Characteristics of Motor Fuel by the Research Method (D 908 - 47 T).

New Tentative Specifications for:

Aviation Gasolines (D 910 - 47 T).

These recommendations were accepted by the Standards Committee on September 4, 1947, and the new tentatives appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

On November 6, the Standards Committee approved the following recommendations submitted by Committee D-2:

New Tentative Method of:

Test for Oxidation Stability of Lubricating Greases by the Oxygen-Bomb Method (D 942 - 47 T),
Test for Oxidation Characteristics of Inhibited Steam-Turbine Oils (D 943 - 47 T),
Test for Penetration of Petrolatum (D 937 - 47 T),
Test for Congealing Point of Pharmaceutical Petrolatums (D 938 - 47 T),
Test for Saponification Number of Petroleum Products by Potentiometric Titration (D 939 - 47 T),
Test for Aromatic Hydrocarbons in Mixtures with Napthenes and Paraffins by Silica Gel Adsorption (D 936 - 47 T),
Measurement of Density of Hydrocarbon Liquids by the Pycnometer (D 941 - 47 T), and
Test for Determination of Purity by Measurement of Freezing Points (D 940 - 47 T).

Revision of Tentative Method of Test for:

Knock Characteristics of Aviation Fuels by the Aviation Method (D 614 - 46 T),
Ignition Quality of Diesel Fuels by the Cetane Method (D 613 - 43 T),
Cone Penetration of Lubricating Grease (D 217 - 44 T),
Sulfur in Petroleum Products by the Lamp-Gravimetric Method (D 90 - 46 T),
Sulfated Residue from New Lubricating Oils (D 874 - 46 T),
Sulfated Residue, Lead, Iron, and Copper in New and Used Lubricating Oils (D 810 - 44 T),
Aniline Points and Mixed Aniline Points of Petroleum Products (D 611 - 46 T), and
Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water (D 665 - 46 T).

Revision and Reversion to Tentative of:

Method of Test for Burning Quality of Kerosine (D 187 - 39), and
Method of Test for Water and Sediment in Petroleum Products by Means of Centrifuge (D 96 - 46).

The new and revised tentatives appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

REPORT OF SUBCOMMITTEE XI ON METHODS FOR DETERMINATION OF SULFATED RESIDUE

Section C of Subcommittee XI during the past several years has been studying methods for the determination of sulfated residue of lubricating oils. These methods have included: (*1*) the direct acid oxidation procedure, formerly designated as A.S.T.M. Emergency Method ES-43 and 43*a*, but more recently designated as A.S.T.M. Method D 874 - 46 T;[1] (*2*) the indirect acid oxidation procedure, published for information;[2] (*3*) the perchloric acid indirect oxidation method; and (*4*) the direct acid oxidation method.

Considerable discussion has arisen regarding whether 650 C. or 775 C. would be the preferable ignition temperature. Cooperative data had been obtained on new and used oils for all of these methods, except the direct acid oxidation method; this method had been tested only on new oils. Therefore, the results of a set of cooperative tests on used oils was completed using two temperatures for the final ignition. These data are tabulated in Table I for ignition at 775 C. and in Table II for ignition at 650 C. The summary of all the data is given in Table III.

Samples S-157, S-158, S-160, and S-164 were synthetically prepared and samples S-166, B-520, and B-529 were used oils. The major metallic constituents in each sample are given at the bottom of Table III.

From the data given in Table III, there is little to choose for the repeatability and reproducibility of sulfated residue determinations by (*1*) A.S.T.M. Method D 874,[1] (*2*) the indirect acid oxidation method, published for information,[2] or (*3*) the direct acid oxidation method, the three preferred methods. However, the error of the methods definitely favors the direct acid oxidation procedure. On the basis of these data Subcommittee XI proposes the changes given below:

1. Retain A.S.T.M. Method D 874[1] as a tentative for the determination of sulfated residue. Delete the last sentence of the scope of this method and insert the following: "This method is not applicable to new or used oils containing lead because of partial loss of lead; sulfated residue from such oils may be determined by A.S.T.M. Method D 810 - 47.[3]"

2. Give no further consideration to the method for sulfated residue which appears as Appendix I of the 1946 D-2 Report.

3. Include direct acid oxidation procedure for sulfated residue as a part of A.S.T.M. Method D 810, Chemical Analysis for Lead, Copper, and Iron in Lubricating Oils,[1] with additional changes as follows:

Title.—Change the title to read "Tentative Method for Sulfated Residue, Lead, Iron and Copper in New and Used Lubricating Oils."

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[1] 1946 Book of A.S.T.M. Standards, Part III-A.

[2] A.S.T.M. Standards on Petroleum Products and Lubricants, December, 1946, p. 13.

[3] 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

Scope.—Revise to read as follows:

1. (*a*) These methods are intended for the determination of sulfated residue, lead, iron, and copper in new and used lubricating oils. Other metallic elements, sulfur, chlorine, and phosphorus, in amounts commonly found in lubricating oils, do not interfere in these methods.

(*b*) The sulfated residue procedure is recommended for use only when a measure of the total metal content is desired. The analysis for sulfated residue is not used when lead, iron, and copper are determined.

(*c*) When sulfated residue is used to indicate the concentration of known metal-containing additives in new oils which do not contain lead, it is recommended that A.S.T.M. Method D 874[1] be used.

TABLE I.—COOPERATIVE SULFATED RESIDUE RESULTS FOR DIRECT ACID OXIDATION METHOD, FINAL IGNITION AT 775 C.

Laboratory	Sulfated Residue, per cent by Weight						
	S-157	S-158	S-160	S-164	S-166	B-520	B-529
Shell	5.93	0.25	3.56	21.2	1.05	1.67	1.57
	5.98	0.27	3.60	21.2	1.17	1.67	1.50
Average	5.96	0.26	3.58	21.2	1.11	1.67	1.54
Phillips	5.57	0.39	3.25	20.30[a]	0.73	1.63	...
	5.98	0.42	3.17	19.86	0.78	1.61	...
	5.55	...	...	20.42	...	1.66	...
Average	5.70	0.41	3.21	20.19	0.76	1.63	...
Socony Vacuum	5.68	0.24	3.32	21.10	0.99	1.83	1.71
	5.50	...	...	...	...	...	...
	5.57	0.21	3.35	21.12	0.93	1.75	1.68
Average	5.58	0.23	3.34	21.11	0.96	1.79	1.70
Texas	...	0.45	3.64	...	0.98	...	...
	...	0.32	...	...	1.05	...	...
	...	0.42	3.60	...	1.11	...	...
	...	0.41	...	...	1.13	...	...
	...	0.39	3.40	...	1.16	...	...
	...	0.33	3.45	...	1.08	...	...
Average	...	0.39	3.52	...	1.09	...	...
Ohio Oil	...	0.36	3.50	...	0.87	...	...
	...	0.36	3.57	...	0.88	...	...
Average	...	0.36	3.54	...	0.88	...	...
Continental	6.32	0.39	3.67	20.82	1.14	1.71	1.61
	6.31	0.39	3.65	20.84	1.12	1.70	1.60
Average	6.32	0.39	3.66	20.83	1.13	1.71	1.61
Sinclair	6.06	0.32	3.46	20.43	1.10	1.67	1.52
	6.21	0.32	3.50	20.47	1.13	1.64	1.51
Average	6.14	0.32	3.48	20.45	1.12	1.66	1.52
General Motors	...	...	3.32	...	0.95	1.76	1.66
	...	...	3.28	...	0.96	1.82	1.69
Average	...	...	3.30	...	0.96	1.79	1.68
Ethyl Corporation	6.34	...	...	21.36	...	1.75	...
	6.36	...	...	21.38	...	1.73	...
Average	6.35	...	...	21.37	...	1.74	...
Average	5.95	0.35	3.46	20.81	1.02	1.71	1.61
Mean Repeatability	0.07	0.07	0.04	0.06	0.03	0.02	0.02
Mean Deviation	0.28	0.06	0.13	0.39	0.10	0.05	0.14
Maximum Deviation	−0.45	−0.14	−0.29	−0.95	−0.29	+0.12	0.11

[a] A 1-g. sample was used.

Respectfully submitted on behalf of Subcommittee XI,

C. M. GAMBRILL
Chairman.

TABLE II.—COOPERATIVE SULFATED RESIDUE RESULTS FOR DIRECT ACID OXIDATION METHOD, FINAL IGNITION AT 650 C.

Laboratory	Sulfated Residue, per cent by Weight			
	S-157	S-164	B-520	B-529
Shell	6.01 5.97 6.18	21.01 21.05 20.90	1.56 1.66 1.65	1.58 1.56 1.50
Average	6.05	21.0	1.63	1.55
Phillips	6.12 6.45 6.25	20.17[a] 20.06 21.02	1.68 1.68 1.69	... ...
Average	6.27	20.42	1.68	...
Socony Vacuum	6.62 6.87 6.62	21.26 21.25 ...	1.95 1.77 ...	1.83 1.84 ...
Average	6.70	21.26	1.86	1.84
Continental[b]	6.73 6.76	20.83 20.89	1.74 1.70	1.73 1.64
Average	6.75	20.86	1.72	1.69
Ethyl Corporation	6.97 6.85	21.42 21.41	1.74 1.72	... ...
Average	6.91	21.42	1.73	...
Sinclair	6.73 6.77	21.21 20.35	1.74 1.71	1.63 1.55
Average	6.75	20.78	1.73	1.59
Average	6.53	20.92	1.71	1.65
Mean Repeatability	0.07	0.17	0.03	0.03
Mean Deviation	0.29	0.33	0.05	0.10
Maximum Deviation	−0.56	−0.86	+0.24	+0.19

[a] A sample was used.
[b] Ignited once.

TABLE III.—PRECISION AND ACCURACY OF SULFATED RESIDUE METHODS.

A—A.S.T.M. ES-43a, 775 C.
B—HNO_3-H_2SO_4 Indirect Acid Oxidation Method, 775 C.
C—$HClO_4$ Indirect Oxidation Method, 775 C.
D—Direct Acid Oxidation Method, 775 C.
E—Direct Acid Oxidation Method, 650 C.

Sample[a]	Repeatability					Reproducibility					Error[b]				
	A	B	C	D	E	A	B	C	D	E	A	B	C	D	E
S-157	0.03	0.03	0.06	0.07	0.07	0.38	0.36	0.22	0.28	0.29	−0.28	+0.12	+0.31	−0.01	+0.57
S-158	0.00	0.01	0.01	0.07	...	0.01	0.01	0.01	0.06	...	+0.03	+0.02	+0.03	+0.09	...
S-160	0.04	0.03	0.05	0.04	...	0.14	0.07	0.08	0.13	...	−0.23	−0.12	−0.17	−0.12	...
S-164	0.2	0.1	0.04	0.06	0.17	1.0	0.3	0.6	0.39	0.33	−2.9	−0.3	−0.2	−0.61	−0.28
S-166	0.02	0.03	0.05	0.03	...	0.06	0.09	0.09	0.10	...	−0.30	−0.17	−0.23	−0.09	...
B-520	0.03	0.01	0.03	0.02	0.03	0.16	0.04	0.07	0.05	0.05	−0.22	−0.07	−0.07	+0.04	+0.04
B-529	0.03	0.03	0.01	0.02	0.03	0.07	0.07	0.09	0.14	0.10	−0.22	−0.08	−0.08	+0.07	+0.11
Average[c]	0.03	0.02	0.04	0.04	0.05	0.14	0.11	0.09	0.13	0.15	0.21	0.10	0.15	0.07	0.24

[a] The major constituents of the samples are as follows:

Sample	Constituents
S-157	cadmium, zinc, phosphorus sulfur
S-158	barium, zinc, phosphorus sulfur
S-160	lead
S-164	lead, bromine
S-166	lead, bromine, iron
B-520	lead, bromine, iron
B-529	lead, bromine, iron

[b] Difference from average Shell direct acid oxidation, 775 C.
[c] Excluding results for sample S-164.

REPORT ON REPRODUCIBILITY AND REPEATABILITY OF THE METHOD FOR DETERMINATION OF BUTADIENE IN POLYMERIZATION GRADE BUTADIENE

OFFICE OF RUBBER RESERVE—COOPERATIVE TEST PROGRAM—BUTADIENE PRODUCTION SAMPLES

PREPARED BY J. C. HILLYER[1]

Under the auspices of the Office of Rubber Reserve, the butadiene producers and the copolymer plant operators carried out a monthly cooperative sample exchange and analysis program on production butadiene samples. Samples were distributed each month to all the plants in the two groups, and each was requested to analyze the samples by all the current official laboratory procedures. Any deviations therefrom or alternate procedures used were to be specifically reported. The determinations were carried out in the plant laboratories by the regular routine operators, who were in many instances nontechnical personnel. Data were compiled by the laboratory distributing the samples and issued through the Butadiene Producers Committee on Specifications and Methods of Analysis.

METHOD OF TEST

The procedure for the quantitative volumetric determination of butadiene, known as the Koppers-Hinckley method, has been in general use in substantially the form prepared for A.S.T.M. consideration beginning with sample 13 in the series and continuing through the last sample, 24. (This method is designated L.M. 2.1.1.9, which differs in no respect for specification grade butadiene from the final procedure L.M. 2.1.1.10). Earlier versions of the method (L.M. 2.1.1.7) received considerable testing, but results are not applicable to the final procedure as submitted to A.S.T.M.

The results obtained using this method have been summarized, and the information is collected in the accompanying tables. Table I lists the cooperating laboratories. In Table II, the results reported, with calculated mean values, deviations, and National Bureau of Standards analyses, are tabulated in full. Table I requires no special comment, except to note that the laboratories listed are all those of the butadiene or copolymer plants operated by the companies listed as agents for Office of Rubber Reserve (formerly Rubber Reserve Co.). They should not be confused with the research laboratories, or other refinery control laboratories of the same companies at the same, or other locations.

In Table II, the results reported by method L.M. 2.1.1.9 by the various laboratories which participated on samples Nos. 13 to 24, inclusive, are tabulated. In all, a total of 28 laboratories participated at one time or another in the testing. For the first few samples, the smaller number of participants resulted from the fact that many laboratories either had not converted their procedure from the earlier version of the method (L.M. 2.1.1.7) or were not equipped with Koppers-Hinckley apparatus and still used the gravimetric test. At the end of the program, shutting down of a few of the plants reduced the active laboratories somewhat. In a few instances on most samples, sample bombs were found to be empty on arrival at the laboratory, and in a similar few instances in each case reports were not received in time to be included in the compilation of data.

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[1] Research Department, Phillips Petroleum Co., Bartlesville, Okla.

TABLE I.—LABORATORIES PARTICIPATING IN OFFICE OF RUBBER RESERVE COOPERATIVE BUTADIENE EXCHANGE ANALYSES.

I. Butadiene Plants—operated by the following as agents for Office of Rubber Reserve:

1. Carbide and Carbon Chemicals Co., Institute, W. Va.[a]
2. Carbide and Carbon Chemicals Co., Louisville, Ky.
3. Cities Service Refining Co., Lake Charles, La.
4. Humble Oil & Refining Co., Baytown, Texas (RuR-SR-10)
5. Humble Oil & Refining Co., Ingleside, Texas
6. Koppers United Co., Monaca, Pa.
7. Neches Butane Products Co., Port Neches, Texas
8. Phillips Petroleum Co., Borger, Texas (RuR-SR-12)
9. St. Clair Processing Corp., Ltd., Sarnia, Ontario, Canada
10. Sinclair Rubber Co., Houston, Texas
11. Shell Chemical Co., Torrance, California
12. Standard Oil Co. of New Jersey, Louisiana Div., Baton Rouge, La., Chemical Products Dept.
13. Standard Oil Co. of California, El Segundo, Calif.
14. Sun Oil Co., Toledo, Ohio

II. Copolymer Plants—operated by the following as agents for Office of Rubber Reserve:

1. Copolymer Corp., Baton Rouge, La.
2. Firestone Tire & Rubber Co., Akron, Ohio
3. Firestone Tire & Rubber Co., Lake Charles, La.
4. Firestone Tire & Rubber Co., Port Neches, Tex.
5. General Tire & Rubber Co., Baytown, Tex.
6. B. F. Goodrich Chemicals Co., Baytown, Tex.
7. B. F. Goodrich Chemicals Co., Louisville, Ky.
8. B. F. Goodrich Chemicals Co., Port Neches, Tex.
9. Goodyear Synthetic Rubber Co., Akron, Ohio
10. Goodyear Synthetic Rubber Co., Houston, Tex.
11. Goodyear Synthetic Rubber Co., Torrance, Calif.
12. National Synthetic Rubber Co., Louisville, Ky.
13. United States Rubber Co., Naugatuck, Conn.
14. United States Rubber Co., Institute, W. Va.
15. United States Rubber Co., Torrance, Calif.

[a] The laboratory at Institute cooperated in these tests, but had a nonspecification apparatus for the butadiene analysis so none of the reported values was used in this tabulation.

NOTE.—In addition, butadiene plant laboratories operated by Lion Oil Co. and Southern California Gas Co. received all samples, but did not report results.

All data reported as obtained by Method L.M. 2.1.1.9 without deviation were used. On the other hand, a few samples were reported calculated on divergent bases, and a few were obtained by reported nonspecification apparatus. These were not included in the table and where they had been included in the averages by the compiling laboratory, the averages have been recalculated. The so-called Torrance Modification employed by Shell Chemical is believed to be identical with L.M. 2.1.1.9 on high-purity butadiene, differing only in application to low-purity material, and results obtained by this procedure were included.

PREPARATION AND DISTRIBUTION OF SAMPLES

The twelve samples circulated covered the usual range encountered in butadiene products. The current specification for polymerization grade is 98.0 per cent by weight as determined by this procedure. Samples ranged from slightly below specification (97.9) to as high as 99.32 per cent. Each sample was submitted to the National Bureau of Standards, and the 1,3-butadiene content was determined by the freezing point method. This determination, with a reported precision of ±0.06 to ±0.08 per cent, serves as a base for analysis of the absolute accuracy of the method as well as its reproducibility. Mass spectrometer analyses were also made by the Bureau. 1,3-butadiene content agreed closely with freezing point values, in most cases being within a few hundredths per cent. Information on the impurities present was of value in studying their effect on reproducibility.

In general, the procedure for supplying the samples was to segregate a sample of production butadiene, usually in a large tank or sphere. This tank was

Sample	No. 13	No. 14	No. 15	No. 16	No. 17	No. 18	No. 19	No. 20	No. 21	No. 22	No. 23	No. 24
Prepared by	Koppers	Humble	Carbide and Carbon	St. Clair Processing	Neches Butane	Standard Oil of N. J., La. Div.	Shell	Cities Service	Phillips	Sinclair	Sun	Standard Oil of Calif.
Testing Laboratory												
Carbide Carbon Institute	98.1	97.9	98.02	98.04	...	97.7	99.0	...	...	...	...	...
Louisville	98.1	98.5	97.8	98.4	...	...	...	98.1	...	...	...	...
Cities Service, Lake Charles	...	...	...	98.4	98.3	98.6	...	98.5	99.3	99.2	98.8	99.1
Copolymer Corp., Baton Rouge	...	...	...	...	98.36	97.7	99.4	97.93	99.3	99.4	98.5	98.9
Firestone Akron	...	...	97.2[a]	98.45	98.14	97.6	99.0	98.3	99.45	99.3	99.3	99.4
Lake Charles	...	...	...	...	...	...	99.26	99.1	99.2	99.2	98.7	99.1
Neches	...	...	...	...	...	...	99.1	98.4	99.2	99.0	98.8	...
General Tire, Baytown	...	...	...	...	...	98.00	99.18	99.19	99.26	99.01	98.99	99.48
Goodrich Borger	...	...	97.75	98.27	98.0	97.90	99.16	98.22	99.11	99.10	98.80	98.85
Louisville	...	...	...	...	...	98.04	...	98.72	...	99.26	98.8	...
Neches	...	98.55	...	...	98.5	98.95	99.0	...	99.3	99.30	98.6	99.07
Goodyear Akron	...	...	...	98.66	98.24	98.27	99.1	98.4	99.10	99.2	98.8	98.7
Houston	...	...	...	98.48	...	98.25	99.46	98.64	99.16	99.31	98.95	99.1
Torrance	...	98.0	98.0	98.26	98.18	97.6	99.07	...	...	99.1	98.76	98.8
Humble Ingleside	...	...	97.9	98.4	97.9	98.1	99.5	...	...	...	...	99.10
Baytown	...	...	...	98.2	98.3	97.9	98.7	98.25	99.9	...	98.94	...
Koppers, Monaca	98.02	98.45	97.83	98.30	98.14	97.99	98.24[a]	98.24	99.2	...	...	...
Nat. Synthetic Rubber, Louisville	...	98.5	98.0	98.5	98.1	98.5	99.4	98.1	99.25	99.0	98.75	98.86
Neches Butane Products, Neches	...	98.7	...	98.3	98.35	98.57	...	98.77	99.35	99.25	97.67[a]	99.0
Phillips, Borger	...	...	...	...	98.2	98.3	99.2	97.9	99.15	99.0	98.3	98.7
St. Clair Processing, Sarnia	97.86	98.48	97.82	98.3	...	98.1	...	98.8	...	99.0	98.85	...
Shell Chemical, Torrance	...	...	...	...	...	...	99.0	...	98.9	99.0	98.7	98.5
Sinclair Rubber, Houston	...	98.4	98.0	98.6	98.1	98.3	99.0	98.5	98.9	99.2	99.0	...
Std. of Calif., El Segundo	...	...	97.7	98.1	98.3	97.9	99.1	98.1	99.2	99.2	98.9	98.9
Std. of N. J., La. Div., Baton Rouge	97.8	98.4	98.0	98.55	98.2	97.9	99.3	98.4	99.4	99.25	99.0	...
Sun, Toledo	97.9	97.9	...	...	98.26	97.8	99.39	98.1	99.36	99.25	98.96	99.01
U. S. Rubber Naugatuck	98.0	...	...	98.2	98.1	97.7	98.6	98.2	98.85[a]	...	99.1	98.9
Torrance	...	...	...	98.45	...	98.5	99.0	98.8	99.2	99.0	98.86	98.9
Number of Samples Averaged	7	11	11	19	18	24	22	23	21	22	23	19
Average Value	97.97	98.34	97.89	98.36	98.20	98.09	99.13	98.42	99.22	99.16	98.83	98.97
Average Deviation from mean	±0.10	±0.22	±0.10	±0.13	±0.11	±0.29	±0.17	±0.28	±0.10	±0.11	±0.15	±0.17
Maximum Deviation from Mean	−0.17	−0.44	−0.19	−0.32	±0.30	+0.85	−0.59	+0.78	−0.32	+0.24	−0.53	+0.51
Nat. Bur. Standards, Freezing Point Method	98.08	98.23	97.90	98.16	98.22	97.93	99.16	98.29	99.32	99.18	98.89	98.73
Nat. Bur. Standards, Mass Spectrometer Method	...	...	...	...	...	...	...	...	...	...	...	...
1,3-Butadiene	98.08	98.16	98.10	98.27	98.27	98.01	99.13	98.22	99.33	99.14	98.83	98.64
1,2-Butadiene	0.0[b]	0.47	0.0[b]	0.5	0.0[b]	0.0[b]	0.52	0.45	0.01[b]	0.0[b]	0.0[b]	0.60
1-Butene	1.66	0.0	0.72	0.59	0.20	0.68	0.22	0.22	0.26	0.15	0.37	0.09
2-Butene	0.6	0.87	1.13	0.15	1.45	0.21	...	0.20	0.38	0.66	0.53	0.51
n-Butane	0.08	0.06	...	...	0.01	0.04	0.07	...	0.01	...	0.05	0.12
Dimer	...	0.06	0.05	0.03	0.04	0.15	...	...	0.02	0.05	0.03	...
Propylene	0.12	0.39	...	0.26	0.03	0.63	...	0.91	...	...	0.06	0.04
Propadiene	...	...	...	...	...	0.28	...	...	...	...	...	...
Pentadienes	...	...	...	...	...	...	...	...	...	...	0.09	...
Other	...	...	Acetone	0.20	...	...	0.06 (C_6H_6)[c]	...	...	...	0.04 (CO_2)	...

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[a] Deviation more than four times the average deviation. Value not included in the average.

[b] Less than 0.1 per cent-limit of precision of the method.

[c] Sample submitted to Nat. Bur. Standards was inadvertantly contaminated with 1.27 per cent acetone. Mass spectrometer and freezing point results have been recalculated on an acetone-free basis.

mixed thoroughly. The sample cylinders were then all filled from this large tank. Alternately, a liquid sample was withdrawn from the storage tank sufficient to supply all the samples, usually into one large cylinder. The contents of this cylinder were thoroughly mixed, and then all the individual sample cylinders were filled from the liquid phase at one time. These sample cylinders were standard Rubber Reserve Scafe-type steel cylinders, bearing a valve at each end. They were cleaned by the individual laboratories and submitted to the laboratory preparing the sample. They were filled by purging, filling liquid full, and drawing off liquid until each contained 2 lb. of butadiene. It was requested that all samples be analyzed on the same date, and in general this was achieved within a few days.

RESULTS OF TESTS

The arithmetic average was obtained for each individual sample. From seven to twenty-four different laboratories cooperated on each. The arithmetic deviation from the mean was calculated in each case. Any result then showing a deviation greater than four times the average deviation was discarded and the average and average deviation recalculated. One result each on samples 15, 19, 21, and 23 was discarded for this reason, as indicated by the note in Table II. A weighted average was obtained for the whole series. This average, representing 220 reported analyses, was ±0.17 per cent. On nine of the twelve samples, average deviation was this good or better, down to ±0.10.

The maximum deviations observed on the samples varied from as low as 0.17 to as high as 0.86 per cent. The weighted average of all the maximum deviation values was ±0.47 per cent, of all positive deviations 0.56 per cent and all negative maximum deviations 0.38 per cent. On this basis, a reproducibility of ±0.20, based on average deviations from the mean, could probably be set for laboratories and operators of the type used. More highly trained operators could probably attain greater reproducibility.

The deviations of the average values from the Bureau of Standards purity by freezing point varied from −0.11 per cent to +0.25 per cent. The arithmetic mean of these deviations, weighted for the number of laboratories reporting each, was ±0.10 per cent. The algebraic sum of the weighted deviations was ±0.05 per cent. This indicates the absolute accuracy is high, and probably close to the limits of error of the freezing point method on which this calculation was based. No consistent error in either direction is definitely indicated. Two samples which were appreciably higher than the National Bureau of Standards values resulted in the positive value for the algebraic sum.

No correlation of the magnitude and direction of error with the impurities shown by mass spectrometer results was possible. The principal impurity present was in most cases normal butene, varying from 0.42 to 2.26 per cent, but no trend in average deviation is noted, nor is there any correlation with the ratio of 1-butene to 2-butene which varied over a wide range. The two samples showing the greatest average deviation and also the greatest (positive) maximum deviation from the mean were both high in propylene. This may be significant, but is not established by so few samples. 1,2-butadiene has been suspected of interference, and it occurred to the extent of approximately 0.5 per cent in five samples. No difference in precision was noted for these compared to the other seven in which this component was less than 0.1 per cent. However, four of these five samples showed a relatively high positive deviation from the absolute value as determined by the freezing point, whereas only one other sample showed an appreciable positive deviation.

TABLE III.—ANALYSIS BY NATIONAL BUREAU OF STANDARDS ON SAMPLE 14, PREPARED BY HUMBLE OIL AND REFINING CO.

One sample withdrawn from the cylinder and five successive determinations made.

Determination	Conjugated diene, per cent by volume
2A	98.17
3A	98.20
4A	98.16
5A	98.17
6A	98.22
Average	98.18
Average deviation	±0.02
Maximum deviation	+0.04

TABLE IV.—REPEATABILITY ON RUBBER RESERVE COOPERATIVE TEST SAMPLE 21.

Distributed by Phillips Petroleum Co.

A. Values reported by Humble Oil and Refining Company, Baytown, Tex. (RuR-SR-10 Laboratory)

99.3 per cent; 99.3 per cent. Average, 99.3 per cent. Deviation, 0.0

B. Values reported by General Tire and Rubber Co., Baytown, Tex.

99.29 per cent; 99.22 per cent. Average, 99.25 per cent. Deviation, ±0.035

C. Values reported by Phillips Petroleum Co., Plains Plant Laboratory, Borger, Tex.

99.16 per cent; 99.18 per cent. Average, 99.17 per cent. Deviation, ±0.01

D. Values reported by National Synthetic Rubber Co., Louisville, Ky.

Operator	Analyses			Maximum Deviations from:		
	1	2	3	Average	Operator Average	Overall Average
A	99.22	99.25		99.235	±0.015	+0.04
B	99.22	99.22	99.25	99.23	+0.02	+0.04
C	99.22	99.22		99.22	0.0	+0.01
D	99.19	99.17	99.22	99.19	+0.03	−0.04
E	99.17	99.17		99.17	0.0	−0.04
Average for 12 analyses				99.21		

The method is based upon the measurement of the unabsorbed, nonbutadiene impurities in the vapor sample. Heavier components which do vaporize are usually absorbed and retained in the maleic anhydride, so the result is always obtained on a dimer, nonvolatile free basis. Calculation of the results back to the basis of the original total sample thus depends also on the precision of the determination of these components and the separation which is made in obtaining the vapor sample for this analysis. Dimer and heavier components were low in all the samples involved in this series. The quantity of heavier components which may be tolerated in the vaporized sample without affecting the value more than 0.10 per cent can be shown to be 5 per cent at 98 per cent purity, since only the effective volume of original sample is changed.

TABLE V.—ANALYSES OF RUBBER RESERVE COOPERATIVE BUTADIENE SAMPLES.

By Plains Plant Laboratory (RuR-SR-12), Phillips Petroleum Co.

Sample	Distributed by	Analyses	Average	Maximum Deviation
No. 17	Neches Butane	98.23; 98.04; 97.71	97.99	−0.28
No. 18	Std. Oil of N. J., La. Div.	98.14; 98.10	98.12	±0.02
No. 19	Shell Chemical	99.19; 99.22	99.205	±0.015
No. 20	Cities Service	98.12; 97.88; 97.93	97.98	+0.14
No. 21	Phillips	99.16; 99.18	99.17	±0.01
No. 22	Sinclair Rubber	99.07; 99.07	99.07	0.00
No. 23	Sun	98.35; 98.34	98.345	±0.005
No. 24	Standard Oil of Calif.	98.82; 98.79	98.805	±0.015

The repeatability is not established by the above values, since each laboratory reported only a single value. In all cases, this was the average of at least two closely agreeing determinations by one operator, and in many cases more tests were made than this. One sample (No. 14) prepared by Humble Oil and Refining Co. was sent to the Bureau of Standards. Its report gave all the individual determinations made on this sample. The data tabulated in Table III show an average deviation from the mean of ±0.02 per cent. This is typical of repeatability by the method.

In Table V, values obtained by the laboratory at Plains Butadiene Plant operated by Phillips Petroleum Co. (No.

8 (I) in Table I) on the various samples analyzed are tabulated. In all but two samples, repeatability was within ±0.03 per cent. Difficulty in checking closely on samples 17 and 20 was experienced, however.

In Table IV, results on those individual determinations, which were reported to Plains Plant on sample 21 which they distributed, are tabulated. Only three laboratories reported their data in this detail. The report from National Synthetic Rubber Co. showing all determinations made by each of five routine operators on the same apparatus is of particular value. The limit of repeatability of ±0.03 per cent claimed for a single operator seems well substantiated, while the variation among five operators gives a repeatability factor on one apparatus of 0.04 per cent.

This report is believed to be an accurate analysis of the data submitted to Office of Rubber Reserve by the participating laboratories. Since the original results submitted to the laboratories compiling results on each sample were not available, reference must be made to their files or reports transmitting such data to the Office of Rubber Reserve concerning the accuracy of this initial step in the analysis of data.

APPENDIX I

PROPOSED RECOMMENDED PRACTICES FOR THE PREPARATION OF NEW TURBINE LUBRICATING SYSTEMS[1]

This is a proposed recommended practice and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Introduction and Scope

1. (*a*) Numerous discussions between the representatives of turbine builders, operators, oil suppliers, turbine equipment suppliers, and consulting turbine engineers in attendance at meetings of Section I on Oil Systems of Turbines, of Technical Committee C on Turbine Oils, of Committee D-2, have indicated the desirability of pooling the available experience with respect to the preparation of new turbine lubricating systems in order that the most desirable practices might be standardized. The pooled information contained herein should be considered as a "guide to desirable practices."

(*b*) It should be emphasized first of all that the preparation of new turbine lubricating systems on large or small units in land or marine service should be accomplished through the cooperative efforts of the turbine builder, the operator, and the oil supplier. No phase of this work should be undertaken without a thorough understanding of the possible effects on subsequent operation of the installation, nor should it be entrusted to persons lacking in experience, without adequate supervision.

(*c*) While it is the primary purpose of this guide to set forth recommendations for the preparation of a system immediately prior to its initial operation, the practices followed during the manufacture and fabrication of the component parts are felt to be so closely related to the operation that a brief summary of procedures found to have given good results is set down as "manufacturers' practices."

MANUFACTURERS' PRACTICES

Piping

2. After fabrication in sections suitable for subsequent assembly, all piping should be thoroughly cleaned by immersion in hot alkaline cleaning solutions for the removal of oil or paint. The same results may be accomplished by an alkaline steam jet cleaning. Following this operation the piping should be thoroughly washed and pickled by immersion in hot dilute hydrochloric or sulfuric acid to which a suitable inhibitor has been added to prevent attack on the bare metal. Where bending sand has been used and may remain in inaccessible parts of the piping, the use of hydrofluoric acid for its removal is recommended. The acid pickling operation should be followed by thorough rinsing for the removal of all acid. The piping should then be dried, after which immediate application of a suitable rust preventive compound should be made. The use of a petroleum-base rust pre-

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[1] This proposed recommended practice is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, October, 1947.

ventive readily soluble in oil is recommended to facilitate subsequent removal from the piping by the method recommended herein or by other methods. All open ends of cleaned and treated pipe should be sealed by a suitable means before storage or shipment. Products used for joint compounds or gasket materials have been known to have a deleterious effect on turbine lubricating oil. Such materials shall be free of acid, lead compounds and oil-soluble components, and other materials adversely affecting oil-service life.

Cast and Fabricated Parts Other Than Tanks or Gear Cases

3. Cast and fabricated parts such as bearing pedestals and caps, bearing brackets, valve gear parts, etc., that are in contact with the oil should be cleaned free from rust and scale by blasting with steel shot or steel grit, wire brushing, or chipping. Rust preventive coatings adequate to supply protection during shipment and storage prior to erection should then be applied.

Oil Tanks and Gear Cases

4. (*a*) Oil tanks, after fabrication has been completed, should be cleaned by steel grit or steel shot blasting or by a steel brush and by a solvent to remove all grease, dirt, and other foreign matter, such as joint and gasket compounds. It should then be protected either by an oil and water-resistant paint or by a rust-preventive coating during shipment and storage prior to assembly.

(*b*) Gear casings, after fabrication has been completed, should be cleaned by steel grit or steel shot-blasting or by a wire brush and by a solvent to remove all dirt, grease, and other foreign matter. It should then be protected by an oil and water-resistant paint or by a rust-preventive coating during shipment and storage prior to assembly.

CLEANING AFTER ERECTION—DIRECT CONNECTED UNITS

General

5. Before any part of the cleaning job is undertaken, a thorough inspection should be made of the entire system by those charged with the responsibility for the cleaning. Its condition should be determined and all peculiarities which may lead to difficulties, such as air pockets or areas of poor drainage, should be noted. This should be followed by an agreement on the procedure to be adopted, which should be concurred in by representatives of the builder, operator, and oil supplier. The initial inspection will indicate the extent of manual labor required before introduction of any oil or flushing fluid. By "manual labor" is meant the removal of thick film (grease-like) rust preventives, brushing, blasting, and sweeping for the removal of welding shot, chips, sand, etc., and general preparation for the flushing procedure which is to follow. In this and all subsequent operations, lintless wiping cloths must be used, as accumulations of lint from this source have been known to contribute to plugging of oilways, with resultant serious damage.

Flushing

6. (*a*) Prior to the installation and circulation of flushing oil, arrangements should be made to prevent the entrance into bearings of any contaminant carried in the oil. This can be accomplished by the installation of blind flanges at the bearing oil inlets with "jumpers" across the bearings so as to avoid long dead ends. On small installations or in other cases where this procedure is not felt to be justified, the bearings' shells may be removed or rotated so as to prevent flow of oil through them, but not in such a manner as to shut off the flow of high-pressure oil. Depending

on the size and layout of the system and the capacity of the pump used to circulate the flushing medium, it may be desirable to sectionalize the piping and flush the sections individually.

(*b*) For those installations in which any rust-preventive materials are present, such materials must be removed before the final charge of lubricating oil is placed in the system. It is the responsibility of the turbine manufacturer to advise the operator or his engineers the type of rust preventive used. Rust preventives which are not readily soluble in turbine oil should be removed prior to assembly. If the rust preventive is oil soluble and can be flushed away, it is desirable to use a flushing oil of the same or lower viscosity than the lubricating oil recommended. If this course is taken, it is considered desirable to use a flushing oil containing the same type of rust inhibitor as the final oil charge. A sufficient volume of flushing oil should be provided in the oil tanks to permit continuous circulation with the auxiliary oil pump. Means should be provided for heating the flushing oil to a temperature of 125 to 180 F. during circulation. Heating to a temperature above the maximum to be expected in service is desirable since it will result in expansion of the piping greater than will occur in service, with consequent more effective loosening of scale and other adhering materials. Continuous use of a filter or centrifuge during the flushing operation is important. Where the lubricating system is not equipped with a purifier, an auxiliary filter can be installed during circulation to remove suspended contaminants which might otherwise be carried in the system. Circulation of the hot flushing oil should be continued for as long a period as necessary; this period may vary from 4 hr. for small units up to 96 hr. or longer for larger units. Following this flushing operation all oil should be drained from the system, particular care being taken to drain all low points in the piping, coolers, and governor mechanism. Heavy solids which have been flushed into the oil reservoir should be removed manually, bearing pedestals inspected and cleaned, and the governor hydraulic mechanism disassembled for the removal of any foreign material.

(*c*) On small systems and those in which no rust-preventive materials have been used, the initial flushing operation can satisfactorily be accomplished with a charge of the same type oil later to be installed as lubricant. In this case the procedure will be similar to that followed with the use of flushing oil, precautions being taken to remove all loose dirt and to prevent the entrance of dirt or other foreign material into bearings and governor parts. Drainage and cleaning of the system in this case should be carried out in the manner described in Paragraph (*b*) for flushing oil.

Displacement

7. (*a*) In certain instances it may be desirable for the complete removal of all flushing oil and any contaminants which it may carry in solution to follow the flushing with a charge of "displacement" oil. The decision on necessity for use of a displacement charge should be left to the judgment of the oil supplier and turbine engineer. Factors to be considered include type and viscosity of flushing oil and general cleanliness of the system. As a displacement oil, the same type of oil as is to be used for the lubricant should be installed in sufficient volume to permit circulation, and the oil, heated to 130 F. to 150 F., circulated for approximately 2 hr. Since the viscosity of this oil will be higher than that of most oils used for flushing, some particles may be suspended and flushed out which were not removed by the less

viscous oil. When inspection of the centrifuge or filter at this time indicates the system to be clean, the blind flanges and jumpers which were initially installed may be removed and circulation continued for an additional 2 to 4-hr. period. During this circulation the governor mechanism should be actuated in order to assure flushing of those parts. When this operation has been completed, where practical, all accessible bearings should be removed and inspected and if found in satisfactory condition the system may be considered clean. During drainage, attention should again be given to all low points and the oil reservoir should be wiped out with lintless cloths and any loose solids removed.

(*b*) In systems in which particular care has been taken during fabrication and erection, it has been found entirely satisfactory to operate the unit for a brief period up to a few weeks on the displacement charge of lubricating oil if laboratory tests indicate that the oil is in satisfactory condition, particularly in regard to viscosity and cleanliness. Where the operating requirements will permit this schedule, the difficulties frequently encountered in the operation of a new system can be worked out before the final lubricating oil is charged.

GEARED AND MARINE SETS

NOTE 1.—The initial remarks made in Sections 2 to 4 with respect to manufacturers' practices will apply generally to direct connected and geared sets alike; however, in view of the greater complexity required in marine systems, some variations in the procedures may be found desirable and the complete procedure described in Sections 8 to 16 has been found to be highly satisfactory by a large number of ship builders when followed closely.

Cleaning Steel Pipes, Valves, and Fittings

8. Before installation and after all fabrication has been completed, all steel pipes, valves, and fittings should be treated as follows and in the order given:

(*a*) All fabricated steel pipes and flanged fittings should be blown with steam or air to remove loose scale and sand. Valves are thoroughly blown and then inspected to insure cleanliness. Valves should not be subjected to the procedures in Paragraphs (*b*) to (*f*).

(*b*) All fabricated steel pipes and flanged fittings should be submerged and washed in a boiling solution of an alkaline metal cleaner such as sodium orthosilicate. The cleansing solution should be prepared in accordance with the recommendations of the manufacturer. Pipes and fittings should be submerged in the boiling solution for a period of approximately 3 to 4 hr. During this operation all dirt, paint, and grease should be removed from the metal, thereby preparing the pipes and fittings for the acid treatment.

(*c*) The pipes and fittings should then be rinsed in warm fresh water.

(*d*) The parts should then be pickled by one of the two following methods, either of which removes all the scale from the metal. The choice of methods depends on the time available for the pickling process:

(*1*) Submerge the pipes and fittings in a solution of dilute hydrochloric acid (Note 2), the composition of which shall be one part of concentrated hydrochloric acid and one part water to which a suitable inhibitor has been added to prevent attack on the bare metal. This process requires pickling for a period of 8 to 16 hr. depending on the condition of the pipes before application of the acid treatment, or

(*2*) In lieu of the dilute hydrochloric acid solution, submerge the parts in an acid bath composed of one part of concentrated sulfuric acid and fifteen parts of water to which a suitable inhibitor has been added to prevent attack on the bare metal. Keep the temperature of the solution between 140 to 180 F. Parts may be pickled in this solution for about 30 to 45 min. Sulfuric acid treatment may be preferred to the hydrochloric acid treatment because of the fact that the former is quicker acting and the same results may be obtained.

NOTE 2.—The experience of some engineers indicates a greater tendency towards corrosion of pipe and fitting pickled with hydrochloric acid solution. It is therefore recommended that wherever convenient, sulfuric acid should be used. If circumstances dictate the use of hydrochloric acid, the time of pickling and time from the acid bath to soda neutralization (Paragraph (*f*)) should be kept to the minimum.

(*e*) Parts should again be rinsed in warm fresh water.

(*f*) Submerge parts in boiling water to which a small amount of sal soda has been added. Rinse with cold fresh water. Dry parts with an air blast.

Protection of Steel Pipes, Valves and Fittings During Storage

9. After the procedures described in Section 7 have been accomplished, the metal has a bright clean finish which is likely to rust. To prevent the rusting of the parts it is necessary to coat the steel pipes, valves, and fittings with a rust preventive for protection during storage prior to installation, as follows:

(*1*) Dip the pickled steel parts in a rust-preventive solution.

NOTE 3.—Suitable oil-soluble rust preventives are preferred, since they will be removed by the flushing oil.

(*2*) After the rust preventive has been applied, carefully seal all open ends of the pipes, valves, and fittings.

Bronze Valves

10. Bronze valves in lubricating oil systems do not require the treatment as outlined in Sections 8 and 9. Bronze valves should be treated as follows:

(*1*) Remove the internal parts of the valves and thoroughly clean the valve bodies.

(*2*) Submerge the valve bodies in boiling water, to which a small amount of sal soda has been added, for approximately 1 hr.

(*3*) Rinse the valve bodies in cold fresh water.

Protection of Main Reduction Gears

11. (*a*) The following practice has been found satisfactory for short-time storage and domestic shipment of gears and gear casings.

(*1*) The gears, journals, bearings, coupling and gear casings should be cleaned with a solvent and allowed to drain and then coated with a rust-preventive compound.

(*2*) If the gears are shipped assembled in the casing, no additional protection is required.

(*3*) If the gears are shipped externally from the case in a crate and supported by the journals, the journals should be given special covering with grease-proof, acid-free paper or fabric wrapper, or both, and additional coatings of rust-preventive compounds.

(*b*) For long-time storage or foreign shipment the following practice has been found satisfactory:

(*1*) Gears, journals, bearings, couplings and gear casings should be given two coatings of a rust-preventive compound. If the gear is shipped separately from the casing in its own crate, the gear teeth as well as the journal should be wrapped with grease-proof, acid-free paper or fabric wrapper, or both, with the wrapper extending down along the rim in order to protect completely the teeth. The wrapper should also be covered with rust-preventive coatings.

(*2*) Some manufacturers prefer to coat reduction gears and the inside of the gear case with heavy grease as a protective material in order to prevent rusting during shipment and storage prior to installation aboard ship.

(*3*) Immediately prior to installation the rust preventive should be removed from the main reduction gears and the inside of the gear case, and the gears then wiped dry with clean lintless cloths.

(*c*) After installation aboard ship a small amount of rust inhibited turbine oil should be poured and brushed over the cleaned surfaces of the main gears and pinions after the gears have been checked for tooth contact. Two electric heaters should be placed in the opposite corners of the sump tank to maintain a uniform temperature throughout the unit and prevent condensation of moisture until the piping is completed and the sump tank is ready for final cleaning.

Cleaning Main Lubricating Oil Sump Tank,

12. (*a*) Where the oil sump is integral with the gear foundation it shall be prepared as follows prior to installation of the gear. (Where the sump is separate from the gear foundation it may be cleaned either before or after installation of the gear.):

(*1*) Grind smooth the entire inside surface of the sump tank, using a flat disk grinder, to remove all scale, roughness, etc. If the tank structure or material of construction does not permit grinding, other suitable means should be employed to insure as complete removal as possible of objectionable materials.

(*2*) Wash the surface of the tank with kerosine or other suitable cleaner to remove all foreign material and then wipe with clean lintless cloths (not waste). Coat all surfaces with rust-inhibited turbine oil. Care shall be taken that all pockets are free from water and foreign material before the system is initially charged.

(*b*) After the installation of the gear and all other parts, the lubricating oil system shall be cleaned as follows:

(*1*) Inspect the sump and gear casing and clean where necessary to remove foreign material. Cover all surfaces with rust-inhibited turbine oil.

(*2*) Remove the two electric heaters mentioned in Section 11(*c*) from the lubricating sump tank.

Cleaning and Flushing Lubricating Oil System

13. After the installation of all parts, the lubricating oil system shall be cleaned and flushed as follows and in the order stated:

(*a*) After the cleaning procedure has been accomplished as described in Section 12, the sump tank is ready to be initially charged with oil.

NOTE 4.—Some shipyards prefer to use regular lubricating oil for the flushing operation, while others prefer the use of a special flushing oil. Some flushing oils contain kerosine and in some instances traces of rust were noted in the pipes and on the gears after using such products. Regular lubricating oil or special flushing oil which contains a rust inhibitor has been satisfactorily used for flushing. It is considered preferable to use flushing oil containing the same type of rust inhibitor as will be used in the final oil charge. When regular lubricating oil is used for flushing, it should be noted that all dirt and foreign material are to be removed from the oil by purifying before the oil is returned to the system.

(*b*) All reduction gear sprays and lubricating oil leads to bearings should be blanked off as near as possible to the parts they serve.

(*c*) To eliminate dead ends in the system, all long leads should be connected by jumpers to the lubricating oil drains.

Jumpers should be of the same approximate diameter as the pipe being flushed.

(*d*) Temporary strainers of monel metal, 40 mesh or finer, or temporary cloth bags should be used on the inside of the lubricating oil service duplex suction and discharge strainers during the flushing period. A temporary strainer box, approximately ¼-in. mesh, should be installed around the lubricating oil suction bell-mouth in the sump tank. Temporary strainers installed prior to the flushing operation should remain in the system until and during the first dock trial. The purifier should be operated continuously during circulation of the flushing oil.

(*e*) The flushing oil should be maintained at a temperature of 125 to 180 F. for the first circulation. The following means for heating the flushing oil may be used. All precautions should be taken to prevent localized overheating of the oil and if a coil or heater is used, it shall have no internal joints:

(*1*) Low-pressure steam (3 to 5 lb. pressure) may be led to the water side of the main lubricating oil cooler,

(*2*) A temporary heating coil may be installed in the lubricating oil sump tank, or

(*3*) A temporary heater may be installed in the piping system.

(*f*) Oil for flushing should be pumped into the sump tanks and circulated through lubricating oil pipes without entering the bearings or reduction gear sprayers at a temperature of 125 to 180 F. for a period of not less than 48 hr. or until no further deposits are removed by the strainers or purifier.

(*g*) Experience in flushing lubricating oil systems has shown that almost all of the larger particles of dirt and foreign material is collected in the temporary strainers during the first 2 hr. of flushing. During this time it is necessary to clean the strainers at frequent intervals of approximately 15 min. The oil purifier should also be inspected frequently and cleaned if necessary.

(*h*) After the flushing operation is completed, the oil should be pumped out of the lubricating oil sump tank and all jumpers removed. The discharge leads to the gear nozzle sprays and turbine bearings should remain blanked off. The main lubricating oil piping system should then be tested under higher than normal working pressures to detect any leaks. The piping layout and cooler design should be checked prior to making this test to assure that no damage will be caused by the test pressures. This test is made by filling the main lubricating oil piping system with turbine oil and bringing the pressure up to 50 psi. gage, or twice the working pressure, whichever is greater. Some shipyards bring the pressure up to 150 psi. for a greater factor of safety. This pressure should be maintained for a sufficient length of time to allow for examination for leaks.

Circulation of Lubricating Oil

14. (*a*) After completing the operations described in Section 13, all blanks should be removed and the pipes adjacent to the blanks that were not flushed previously cleaned with lintless cloths (*not waste*) and reassembled.

(*b*) The lubricating oil sump tank should be inspected and cleaned, if necessary, the final charge of lubricating oil then pumped into the system.

(*c*) Prior to any operation of the turbines and gears, lubricating oil should be circulated through the complete system at a temperature of approximately 130 F. with sprays in operation and with bearing supply needle valves wide open for approximately 48 hr. or until no further solids are removed by the purifier or strainers. Strainers and purifier should be cleaned periodically if necessary. Inspection plates on the reduction gears are then removed and sprayers and gear teeth inspected.

(*d*) After the circulation described in Paragraph (*c*), a representative group of turbine bearings should be examined and if found dirty, a further inspection of bearings made and all dirty bearings cleaned. At this time the lubricating oil in the sump tank is purified by the lubricating oil purifier.

(*e*) Circulation of lubricating oil shall again be started. During this period the jacking gear shall be engaged and the main units turned over for a period of at least 2 hr. The purifier should be operated during this time.

(*f*) After this circulation, a representative turbine bearing and pinion bearing should be examined and if they are not found to be clean, inspection of all bearings should be made.

(*g*) After the first dock trial, the oil in the lubricating oil sump tanks should be transferred to the lubricating oil settling tanks, and the lubricating oil sump tanks cleaned with lintless cloths (*not waste*).

(*h*) All temporary strainers should be removed.

(*i*) After the lubricating oil is allowed to settle, it should be run through the lubricating oil purifier before it is returned to the main lubricating oil sump tank.

Inspections

15. After the first dock trial, the following inspections should also be made:

(*1*) Lubricating oil sump tank,

(*2*) Representative bearings, including turbine thrust bearing,

(*3*) Main thrust bearing,

(*4*) Gear surfaces and sprayer nozzles (through the inspection doors) and couplings and oil strainers,

(*5*) Inlet end of lubricating oil coolers, and

(*6*) Representative piping sections between strainers and bearing, paying particular attention to surfaces of the steel piping and internal parts of oil-actuated governor or controls.

Additional Factors

16. While proper construction and preparation of the system prior to operation are essential, there are a number of additional factors involved in obtaining continuous trouble-free operation of the turbine unit. These include adequate facilities for oil maintenance, proper use of such facilities, and means adopted for periodic checking of oil quality. It may be possible to make recommendations in regard to these factors at some later date.

APPENDIX II

PROPOSED METHOD OF TEST FOR EVAPORATION LOSS OF LUBRICATING GREASES AND OILS[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method describes a procedure for the determination of evaporation loss of lubricating greases or low-viscosity oils designed for low-temperature service.

Outline of Method

2. The sample of lubricant in a closed evaporation cell is placed in a bath maintained at 210 F. Heated air is passed over its surface for 23 hr. The evaporation loss is calculated from the loss in weight of the sample.

Apparatus

3. (*a*) *Evaporation Cell,* as described in the Supplement.

(*b*) *Air Supply System,* capable of supplying to the cell the required flow of air free of mechanically entrained particles. A 16-in. length of 1-in. diameter pipe packed with glass wool has been found satisfactory for filtering the air.

(*c*) *Oil Bath,* as described in the Supplement.

(*d*) *Thermometer.*—An A.S.T.M. Saybolt Viscosity Thermometer having a range of 204 to 218 F. and conforming to the requirements for thermometer 22F – 39 as prescribed in A.S.T.M. Standard Specifications E 1.[2]

Standardization of Apparatus

4. Immerse the cell to the level shown in Fig. 1 in the bath operating at 210 ± 1 F. Insert in the vent a small rubber stopper carrying a tube leading to a calibrated gas meter. Adjust the air pressure to produce a flow through the cell and meter equivalent to 2 liters per min. at standard temperature and pressure. Observe and record the pressure necessary to produce this flow.

Procedure for Greases

5. (*a*) Weigh the clean sample cup and hood to the nearest milligram. Remove the hood and fill the cup with sample, taking care to avoid occlusion of air. Smooth the surface level with the rim of the cup with a straight-edged spatula. Thread the hood tightly onto the cup without disturbing the smoothed grease surface. Weigh the assembly and record the net weight of the sample to the nearest milligram.

(*b*) With cover in place, but without the hood and sample cup attached, allow the evaporation cell to acquire the temperature of the bath, 210 ± 1 F., by immersing the cell in it, as shown in Fig. 1. Allow the cell to remain in the bath at least ½ hr. before beginning the test. During this period, allow clean air to flow through the cell at the prescribed rate, 2 liters per min., as indicated by the gage pressure. Shortly before introducing the sample, temporarily connect the gas meter to the vent on the cell cover and make sure that the proper air flow is indicated (Note). Then remove the cover, thread the weighed hood and sample cup into place, and replace the cover. Pass clean air through the cell for 23 hr. ± 5 min.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, October, 1947.

[2] 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

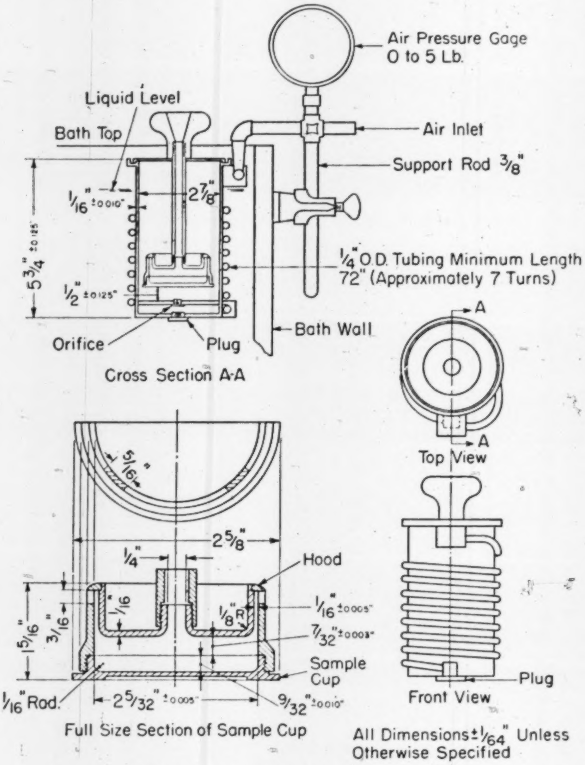
Air Pressure Gage
0 to 5 Lb.
Liquid Level
Bath Top
Air Inlet
Support Rod ⅜"
1/16" ±0.010"
2⅞"
5¾" ±0.125"
½" ±0.125"
¼" O.D. Tubing Minimum Length
72" (Approximately 7 Turns)
A
Orifice
Plug
Bath Wall
Cross Section A-A
A
Top View
5/16"
2⅝"
¼"
Hood
1/16"
1/8" R
1/16" ±0.005"
7/32" ±0.005"
15/16"
3/16"
Sample
Cup
Plug
1/16" Rod
2 5/32" ±0.005"
9/32" ±0.010"
Front View
Full Size Section of Sample Cup
All Dimensions ± 1/64" Unless
Otherwise Specified

FIG. 1.—Evaporation Test Cell.

NOTE.—If the gas meter indicates a rate perceptibly different from 2 liters (standard temperature and pressure) per min., it is likely that the orifice has become fouled and should be cleaned. Backwashing with chloroform will generally dislodge any obstruction; if necessary the orifice should be removed and cleaned.

(*c*) At the end of the 23-hr. period, remove the assembled sample cup and hood from the cell and allow to cool to room temperature. Again check the flow rate with the gas meter as further assurance against fouling of the orifice during the test. Determine the net weight of the sample to the nearest milligram.

Procedure for Oils

6. (*a*) Weigh the clean sample cup and hood to the nearest milligram. Trans-

fer, by means of a pipet, 10.00 ± 0.05 g. of sample to the cup. Assemble the cup and hood, being careful not to splash oil on the underside of the hood. Weigh the assembly and record the net sample weight to the nearest milligram.

(*b*) Evaporate the sample as described in Section 5 (*b*) and (*c*).

Calculation

7. Calculate the evaporation loss of the sample by means of the following equation:

Evaporation loss, per cent by weight =

$$\frac{S - W}{S} \times 100$$

where:

S = initial weight of sample, in grams, and

W = weight of sample, in grams, after the test.

Precision

8. (NOTE.—Limits of precision are being developed from results of cooperative tests.)

SUPPLEMENT

APPARATUS

Apparatus:

A1. *Evaporation Cell*[3] and attachments conforming with the dimensional tolerances indicated in Fig. 1 and capable of being supported upright in the oil bath. Other structural details are as follows:

(*a*) The body and cover of the cell shall be constructed of tinned brass and the air heating coil of tinned copper tubing.

(*b*) The sample cup, hood, eduction tube, and orifice shall be constructed of 18 per cent chromium, 8 per cent nickel alloy steel. A suitable material is an alloy steel conforming to grade S, type 304, of A.S.T.M. Specifications A 240, for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels.[4] To facilitate removal and separation of the cup and hood for inserting the sample and weighing, the sample cup shall be threaded to the hood and this in turn to the eduction tube of the cover. The orifice shall be made removable.

(*c*) The cover of the cell shall be made air-tight.

A2. *Oil Bath* of sufficient depth to allow submersion of the evaporation cell to the proper level and capable of being controlled at 210 ± 1 F. with a maximum variation throughout the bath of 1 F. Circulation of the oil heating medium by a pump or stirrer is recommended. Sufficient heat capacity shall be provided to return the bath to the required temperature within 60 min. after immersion of the cell. The bath shall be provided with a temperature well such that the 206-F. point of the Saybolt Viscosity Thermometer is at the same level as the upper surface of the bath cover. The bath shall be arranged so that there are no drafts or wide fluctuations in temperature around the evaporation cell.

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[3] The use of the Precision Scientific Co. test cell, Catalog No. 74914, gives the same results as the apparatus described in section A1. Where the prescribed equipment is not available, the Catalog No. 74914 equipment may be used. However, greater care in the handling of this test cell is required because of the less substantial construction.

[4] 1946 Book of A.S.T.M. Standards, Part I-A.

APPENDIX III

PROPOSED METHOD FOR DETERMINATION OF BUTADIENE CONTENT OF POLYMERIZATION GRADE BUTADIENE BY KOPPERS-HINCKLEY METHOD[1,2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method is intended for the determination of butadiene in polymerization grade butadiene. It is applicable to other mixtures of C_4 and lighter hydrocarbons containing 95 per cent or more butadiene. Water and acidic constituents must be removed before analysis. Materials boiling above the C_4 range must be removed if they vaporize under the test conditions but do not interfere if they remain in liquid form. C_4 alkylacetylenes do not affect the results.

NOTE 1.—This method is also applicable, with less accuracy, to recycle grade butadiene and other mixtures containing smaller percentages of butadiene.

Outline of Method

2. A measured volume of sample is swept by a stream of carbon dioxide through molten maleic anhydride into a scrubber and measuring buret containing a solution of potassium hydroxide. 1,3-butadiene is chemically absorbed in maleic anhydride, while the continuous stream of carbon dioxide prevents the physical absorption of nonreactive gases. The carbon dioxide is removed during passage through the potassium hydroxide solution so that the difference between the original volume of sample and the residual volume, corrected for the vapor pressure of the potassium hydroxide solution, is a measure of the butadiene content of the sample.

Apparatus

3. The apparatus shall be assembled as shown in Fig. 1, using glass or metal capillary tubing for interconnecting the component parts which are described in Paragraphs (*a*) to (*g*). Connections to the tubing may be made conveniently with interchangeable spherical joints sealed with hydrocarbon insoluble stopcock lubricant or thermoplastic; rubber shall not be used.

(*a*) *Sample Pipet.*—A water-jacketed glass bulb connected through capillary tubing, sealed to each end, to a special reversing stopcock as shown in Fig. 2. The total volume of the bulb and capillary tubing shall be 100 ± 0.1 ml. The water jacket shall be designed to permit circulating water through it.

(*b*) *Maleic Anhydride Scrubber.*—A steam-jacketed glass tube, conforming to the dimensions shown in Fig. 3, with a water-cooled condenser inserted in a standard-taper glass joint at the top and

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, October, 1947.

[2] This method was developed by Dr. John Hinckley on a Koppers Fellowship at the Mellon Institute of Industrial Research and submitted to the Butadiene Producer's Committee on Specifications and Methods of Analyses of the Office of Rubber Reserve. It has been cooperatively tested by them and appears in the Butadiene Laboratory Manual, Office of Rubber Reserve, as Method No. 2,1,1,10.

a sintered glass disk of medium porosity sealed near the lower end to which a stopcock is attached to permit draining.

(c) *Steam Generator.*—Any suitable steam generator. A convenient generator consists of a glass flask, containing two carbon electrodes, in which steam is generated from a very dilute solution of electrolyte.

(d) *Potassium Hydroxide Scrubber.*—A bulb of chemically resistant glass, constructed as shown in Fig. 4, with a spiral tube sealed in it, a side arm for connection to a leveling bulb, and a capillary extension ending in a three-way stopcock.

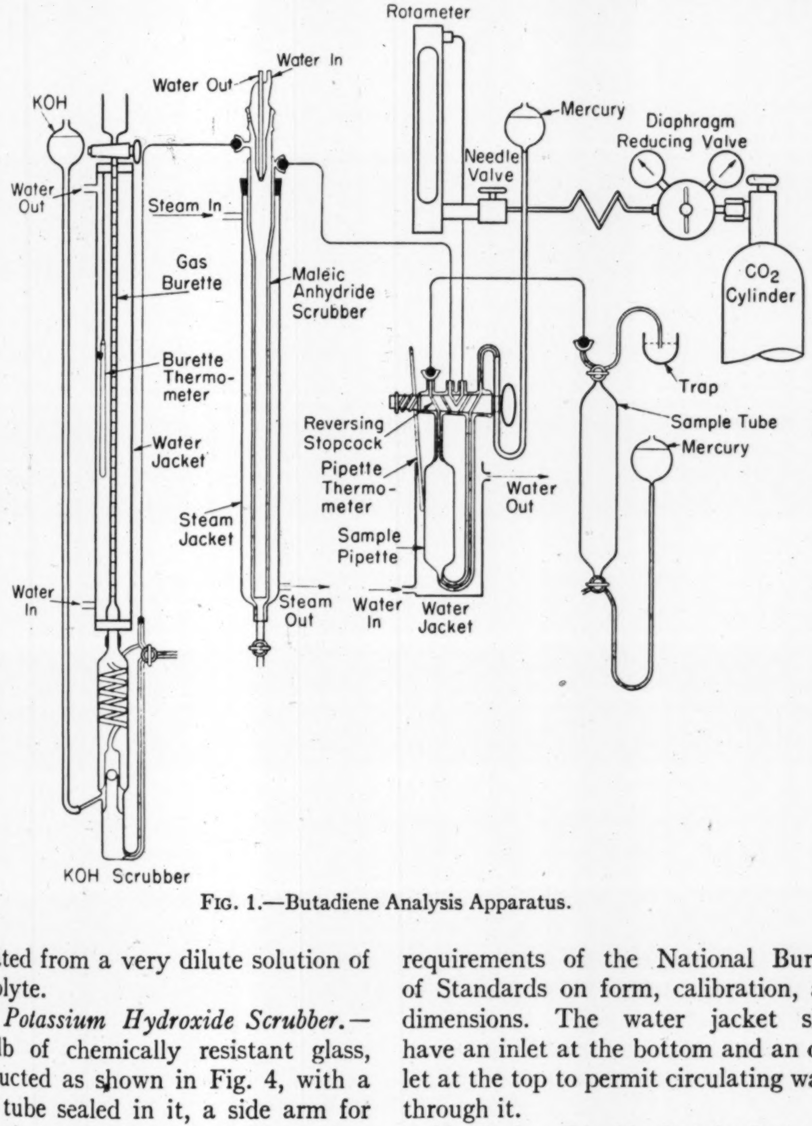
Rotameter
Water In
Water Out
KOH
Mercury
Diaphragm
Reducing Valve
Needle
Valve
Water
Out
Steam In
Gas
Burette
Maleic
Anhydride
Scrubber
CO2
Cylinder
Burette
Thermo-
meter
Trap
Reversing
Stopcock
Sample Tube
Mercury
Water
Jacket
Pipette
Thermo-
meter
Water
Out
Steam
Jacket
Sample
Pipette
Water
In
Steam
Out
Water
In
Water
Jacket
KOH Scrubber

FIG. 1.—Butadiene Analysis Apparatus.

(e) *Gas Buret.*—Any calibrated water-jacketed gas buret graduated in 0.1 ml. over a volume of approximately 30 ml. and having not less than 30 mm. between milliliter graduation marks. The buret shall be of alkali resistant glass and shall conform to the general requirements of the National Bureau of Standards on form, calibration, and dimensions. The water jacket shall have an inlet at the bottom and an outlet at the top to permit circulating water through it.

(f) *Carbon Dioxide Supply System.*—A cylinder of carbon dioxide (treated as described in Section 4 (*d*)), a 2-stage reducing valve with a gage capable of delivering gas at a pressure of approximately 10 psi. constant to ± 0.5 psi., a needle valve, and a flowmeter calibrated in the range of 0 to 60 ml. per min. at 760 mm. of Hg.

(*g*) *Thermometers.*—Mercury-in-glass thermometers having a length of 10 to 15 in. and a range of 0 to 40 C. and graduated in 0.1 to 0.2 C.

Reagents and Materials

4. (*a*) *Maleic Anhydride,* having a minimum melting point of 52 C. Purification by vacuum distillation is necessary if the melting point is below 52 C.

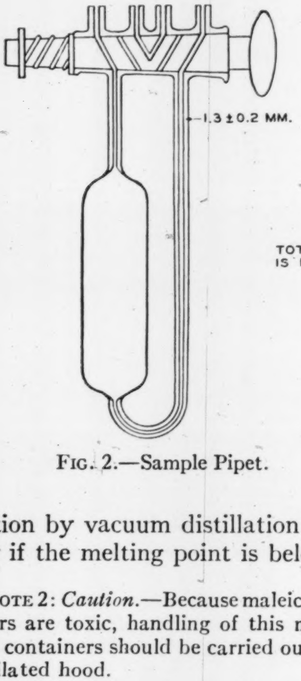

FIG. 2.—Sample Pipet.

NOTE 2: *Caution.*—Because maleic anhydride vapors are toxic, handling of this material in open containers should be carried out in a well ventilated hood.

(*b*) *Diamylamine* (*c.p.*).

(*c*) *Potassium Hydroxide Solution* (45 ± 2 per cent by weight).—Prepare by dissolving 1000 g. of c.p. KOH in 890 ml. of distilled water. Determine the total alkalinity, expressed as percentage of KOH by weight, by titration of a weighed quantity of the solution with standard acid, using methyl orange as indicator. Adjust the strength of the solution to 45 ± 2 per cent by addition of water or KOH.

(*d*) *Carbon Dioxide* of such purity that not more than 0.01 ml. of gas is collected in the gas buret in 30 min. of flow through the assembled apparatus under the test conditions (Section 6(*e*)). Carbon dioxide of this purity may be prepared from carbon dioxide supplied from commercial liquid cylinders or from solid carbon dioxide.

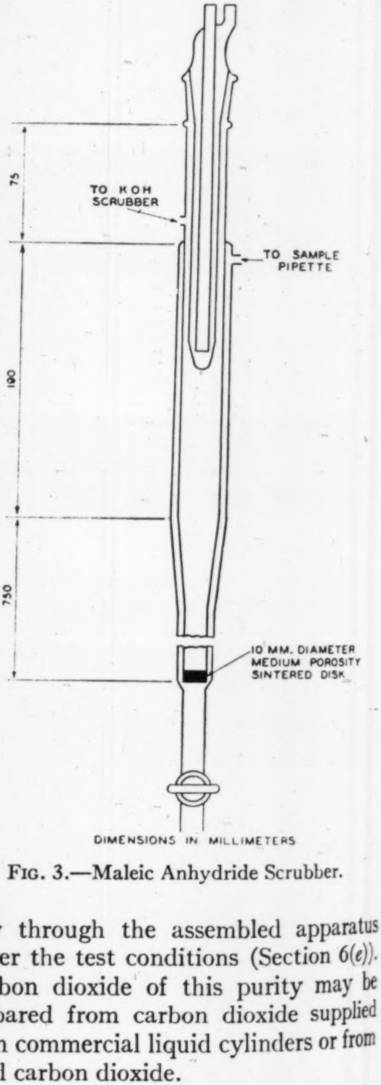
75
TO KOH SCRUBBER
TO SAMPLE PIPETTE
190
750
10 MM. DIAMETER MEDIUM POROSITY SINTERED DISK
DIMENSIONS IN MILLIMETERS

FIG. 3.—Maleic Anhydride Scrubber.

If a liquid cylinder is used, place it on a scale and vent, as rapidly as possible.

70 to 75 per cent of the contents. The venting must be sufficiently rapid so that, during the latter stages, frost collects on the lower part of the cylinder. This process will reduce the impurities from an initial value of a few tenths of a per cent to a few parts per million.

If solid carbon dioxide is used, purify it by venting from a packed container which may be either a heavy walled glass flask or a cylindrical pressure vessel of suitable alloy (Note 3). If a heavy walled glass flask (about 4-liter capacity) is used, it shall be equipped with a mercury pressure release, having a mercury height equivalent to 8 to 10 psi. pressure, and shall be surrounded with an insulated box. Pack the flask with finely crushed solid carbon dioxide and vent until (about 30 to 40 min.) the impurities are sufficiently reduced. If a cylindrical pressure vessel (about 5-liter capacity) is used, it shall be tested to withstand a pressure of 2000 psi. and shall be equipped with a valve and bursting disk and a removable cover. Fill the vessel, while tamping, with finely crushed solid carbon dioxide. Then close it securely and place it on a scale. Vent the gas intermittently without allowing the pressure to exceed 100 psi. until the weight of carbon dioxide is not more than 60 per cent of the weight of water required to fill the vessel.

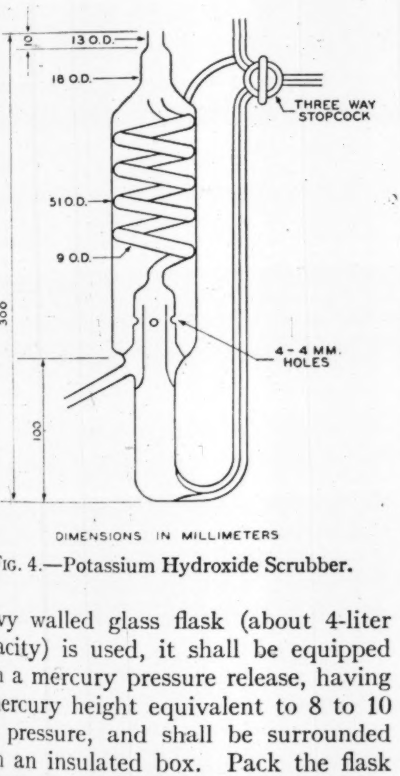

FIG. 4.—Potassium Hydroxide Scrubber.

NOTE 3.—Vessels of ordinary carbon steels, especially rimmed or free machining steel, are not suitable because the metal becomes brittle on cooling to low temperature. Therefore, if a cylindrical pressure vessel is used, it must be constructed of a safe material such as an austenitic steel or a nickel steel suitably heat treated.

(e) *Stopcock Lubricant.*—A rubber-free stopcock lubricant that is resistant to hydrocarbons, for use on stopcocks and joints in contact with hydrocarbons, and a water insoluble lubricant for use on stopcocks in contact with potassium hydroxide solution.

(f) *Mercury.*—Clean, dry mercury.

Samples

5. All liquid butadiene samples from various sources (such as spheroid pressure tanks, pipe lines, tank cars, cylinders, and sample bombs) shall be sampled from the liquid phase. If stored, keep liquid samples in clean, dry steel cylinders having a working pressure of 100 psi. or more.

Preparation of Apparatus

6. (a) *Cleaning Sample Pipet.*—Clean the sample pipet, whenever there is the slightest evidence of grease on the walls, by flushing with benzene. Remove the benzene by evaporation with a stream of air. After this treatment, check the lubrication of the stopcock. Usually, if the stopcock is not turned until the benzene is completely removed and if it is turned slowly the first time after flushing, relubrication will not be necessary.

(*b*) *Assembly of Apparatus.*—Assemble the apparatus as shown in Fig. 1 and carefully lubricate stopcocks and joints and tighten all connections to ensure that the system is free of leaks. Insert thermometers in the water jackets of the gas buret and sample pipet. Attach the water inlets and outlets of these jackets to a thermostated water circulating system controlled so that the buret and pipet are maintained at a temperature constant to plus or minus 0.1 C. between 20 and 40 C. or, preferably, a few degrees above room temperature.

(*c*) *Preparation of Potassium Hydrooxide Scrubber and Gas Buret.*—Introduce 10 ml. of mercury through the stopcock of the potassium hydroxide scrubber. Lower the leveling bulb connected to the side arm of this scrubber and introduce a sufficient quantity of potassium hydroxide solution to fill the buret and scrubbing assembly. Replace approximately 40 ml. of the solution with fresh potassium hydroxide after each analysis. Pour 40 ml. of the fresh solution into the cup at the top of the gas buret, open the stopcock, and allow the solution to be drawn slowly down into the buret. Remove the excess used solution from the leveling bulb.

(*d*) *Preparation of Maleic Anhydride Scrubber.*—Start the steam generator and heat the maleic anhydride scrubber. Introduce 65 g. of maleic anhydride and 1.5 ml. of di-*n*-amylamine, either mixed or simultaneously. Replace the maleic anhydride after 25 determinations or every three days, whichever occurs first. Preliminary treatment of fresh anhydride or of anhydride previously used on a different type of sample may be necessary (see Note 5).

(*e*) *Flushing with Carbon Dioxide.*—Adjust the pressure of the reducing valve of the cylinder of purified carbon dioxide to about 10 psi. Adjust the needle valve so that the flow rate of the carbon dioxide is as rapid as possible without producing slugging or the formation of large bubbles in the maleic anhydride scrubber. Continue flushing until not more than 0.01 ml. of gas is collected in the buret in 30 min.

Procedure for Introduction of Sample

7. (*a*) While the carbon dioxide is passing into the scrubber system through the short by-pass drilled in the plug of the reversing stopcock, introduce the sample (previously passed through a soda - asbestos (Ascarite) drying tube to remove water and acidic constituents if necessary) into the sample pipet.

(*b*) If the sample can be readily transferred to a sample tube of the type shown in Fig. 1, its use is convenient. In this case, introduce mercury into the sample pipet from a leveling bulb attached to the outlet tube farthest to the right on the reversing stopcock, permitting the air which is displaced from the pipet to escape through the outlet farthest to the left on the reversing stopcock and through the three-way stopcock at the upper end of the sample tube. Completely displace the air in the sample pipet and connecting lines and allow a few drops of mercury to flow out. Then, by manipulation of the leveling bulb containing mercury attached to the lower stopcock of the sampling bulb, displace the mercury from the sample pipet with the sample. Finally, release the excess pressure of the sample in the pipet by venting to the atmosphere through the three-way stopcock at the top of the sample tube and a trap, containing mercury or salt water, to prevent the possibility of diffusion of air into the pipet. Read and record atmospheric pressure.

(*c*) If the gas to be analyzed is available in a container under pressure, connect the container directly (or through a soda-asbestos (Ascarite) drying tube if necessary) and let sufficient sample flow through the pipet to displace all air from it. Finally, stop the flow of sample and adjust the pressure in the pipet by venting to the atmosphere. Read and record atmospheric pressure.

Procedure for Analysis of Sample

8. After the system has been flushed sufficiently (Section 6(*e*)) turn the reversing stopcock through 180 deg. (Note 4) to sweep the sample with carbon dioxide into the maleic anhydride scrubber and the residual gas through the potassium hydroxide scrubber for removal of carbon dioxide to the gas buret. Throughout the determination, keep the surface of the potassium hydroxide in the leveling bulb at a point corresponding to the zero mark on the gas buret. Read the buret at intervals until (normally 20 to 40 min.) constant readings are obtained. Then lower the leveling bulb so that the surface of the potassium hydroxide solution in it is level with the meniscus in the buret and take the final reading (Note 5). Read and record the temperature of the water in the jacket of the gas buret.

TABLE I.—VAPOR PRESSURE OF KOH SOLUTION.

TEMPERATURE, deg Cent.	PRESSURE, Mm. of Hg.
20	4.0
21	4.2
22	4.5
23	4.8
24	5.1
25	5.4
26	5.8
27	6.2
28	6.6
29	7.0
30	7.4
31	7.9
32	8.4
33	8.9
34	9.4
35	10.0
36	10.6
37	11.3
38	11.9
39	12.6
40	13.4

NOTE 4.—After a period of about 15 min., the sample will be completely swept out of the pipet and the reversing stopcock may be turned so that the carbon dioxide stream by-passes the pipet. The introduction of another sample (Section 7) during the period required to complete the analysis will reduce the time required per determination.

NOTE 5.—The first analysis made with fresh maleic anhydride or with maleic anhydride previously used on a sample of appreciably different butadiene content may be in error. Therefore, in such cases, it is necessary to run several determinations until reproducible results are obtained.

Calculations

9. (*a*) Calculate the volume (mole) percentage of butadiene in the sample as follows:

Butadiene, per cent by volume =

$$100\left[1 - \frac{V_b}{V_p}\left(1 - \frac{P}{A}\right)\right]$$

where:

V_b = volume in milliliters of the gas in the buret (Section 8),

V_p = volume in milliliters of the sample pipet,

P = vapor pressure in millimeters of mercury of the KOH at the buret temperature (see Table I), and

A = atmospheric pressure in millimeters of mercury.

(*b*) Calculate the weight percentage of butadiene in the sample as follows:

Butadiene, per cent by weight =

$$\frac{2.228V(100 - R)}{100D}$$

where:

V = volume (mole) percentage of butadiene in the sample,

D = density of the sample in grams per liter at 30 C. and 760 mm. of mercury (if the density is not known, D may be assumed to be equal to 2.228), and

R = weight percentage of nonvolatile materials (such as styrene, dimer, and other nonvolatile residues) in the sample. The percentage of these materials, if present, should be determined by auxiliary methods.

If the sample consists essentially of a mixture of butenes and butadiene (containing at least 70 per cent butadiene), calculate the weight percentage of butadiene as follows:

Butadiene, per cent by weight =

$$(V - K) \times \frac{100 - R}{100}$$

where K = value read from Fig. 5.

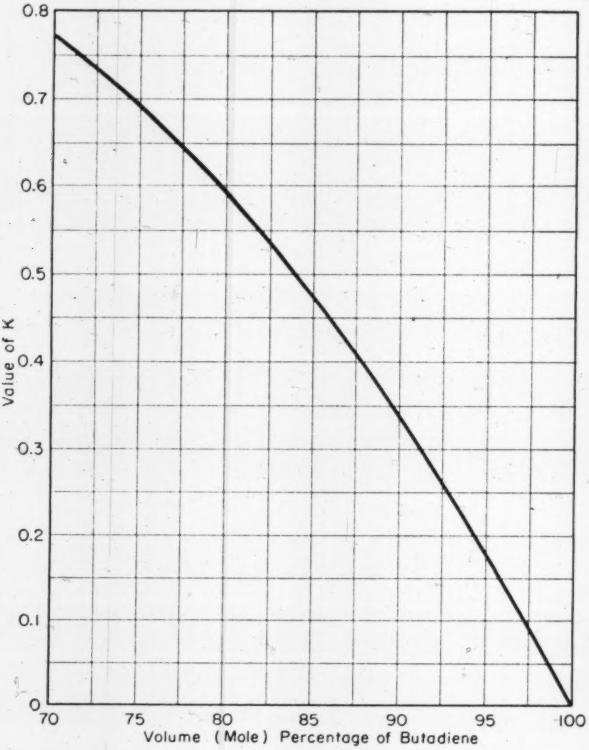

FIG. 5.—Values of K for Butadiene - Butene Mixtures.

Precision

10. Results should not differ from the mean by more than the following amounts:

Repeatability, One Operator and Apparatus	**Reproducibility,** Different Operators and Apparatus
0.05 per cent of mean	0.2 per cent of mean

APPENDIX IV

PROPOSED METHOD FOR DETERMINATION OF COLOR INDEX OF PETROLEUM PRODUCTS BY PHOTOELECTRIC COLORIMETER[1]

This is a proposed method and is published as info mation only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

HISTORICAL BACKGROUND OF PHOTOELECTRIC COLORIMETRIC METHOD

Since 1940, Subcommittee VI on Color, of Committee D-2 on Petroleum Products and Lubricants, has been studying the application of the photoelectric colorimeter to the measurement of the color of lubricating oil. The work was motivated by a combination of the uncertainty of the human eye, the lack of supply of satisfactory Union colorimeter glass color standards, and an attempt to obtain a method with a more basic foundation.

The Proposed Method of Test for Color of Lubricating Oil by Means of Photoelectric Colorimeter, which was published as Appendix II of the 1942 Report of Committee D-2,[2] was the initial procedure employed in the study. This method specified in general terms a photoelectric colorimeter that met prescribed requirements. Standardization was accomplished by the use of six aqueous solutions.

The instrument requirements, as written in the method, limited the selection of photoelectric colorimeters to a very small percentage of the instruments available. In order that more instruments could be utilized by a photoelectric color method for petroleum products, various changes were studied which resulted in liberalization of instrument requirements and certain changes in procedure. This Proposed Method for Determination of Color Index of Petroleum Products by Photoelectric colorimeter differs basically from the previous method in procedure as follows:

1. Glass color filters of known spectral characteristics and with National Bureau of Standards certified values are used for calibration instead of the aqueous solutions.
2. A calibration curve for each instrument is required, and it is constructed by plotting the differences between readings obtained with the standard glasses and the certified values of the same glasses.
3. Corrections from calibration curves are applied as corrections to readings obtained on petroleum products.
4. Requirements are established to control instrument sensitivity and response.

The relationship between A.S.T.M. Union color and color indices by this proposed photoelectric method is approximately the same as experienced in the previous proposed method.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, October, 1947

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 317 (1942).

Scope

1. This method is intended for the photoelectric determination of a color index of petroleum products. It is designed particularly as a method for determining compliance with specification requirements for color characteristics of lubricating oils and products having similar spectral properties. It is not applicable to samples that show a turbidity at room temperature or to materials whose spectral properties differ markedly from sample to sample.

NOTE 1.—The color index gives a reasonably satisfactory correlation with results obtained by present A.S.T.M. visual methods applied to petroleum products. Figure 1 may be used to convert color index values to corresponding A.S.T.M. Union color numbers (A.S.T.M Method D 155, Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter[2a]) with an uncertainty of approximately one half of an A.S.T.M. color number in most cases.

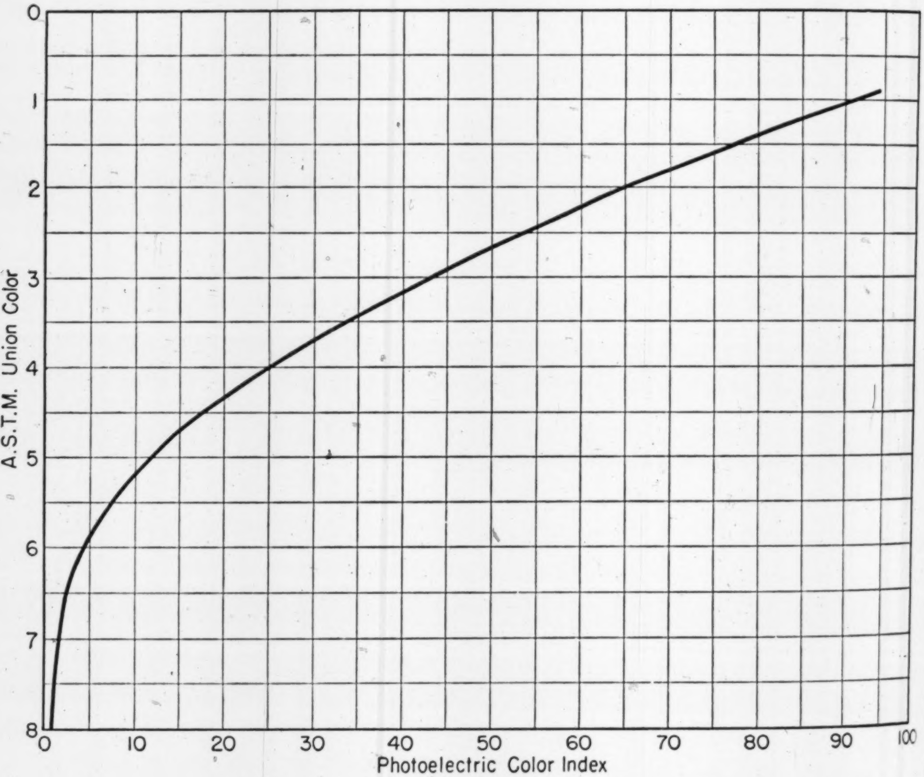

Fig. 1.—Correlation of Photoelectric Color Index with A.S.T.M. Union Color Number. Conversion from photoelectric color index is generally accurate within one half of an A.S.T.M. Union Color number.

Definitions

2. (a) *North Sky Transmittance.*—The corrected instrument response for a 20-mm. layer of sample, expressed in percentage transmittance, relative to that for distilled water and found by use of the north sky filter specified in Section A3 of the Supplement.

(b) *Violet Transmittance.*—The corrected instrument response for a 20-mm. layer of sample, expressed in percentage transmittance, relative to that for distilled water and found by use of the violet filter specified in Section A3 of the Supplement.

[2a] 1946 Book of A.S.T.M. Standards, Part III-A.

(*c*) *Red Transmittance.*—The corrected instrument response for a 20-mm. layer of sample, expressed in percentage transmittance, relative to that for distilled water and found by use of the red filter specified in Section A3 of the Supplement.

(*d*) *Color Index.*—Color index is the numerical value of the north sky transmittance for values from 3.0 to 95.0 per cent inclusive. For north sky transmittance values above 95.0 per cent, color index is equal to ninety plus one tenth of the violet transmittance. For north sky transmittance values below 3.0, the color index is a value calculated from the north sky transmittance of a solution of one part by weight of sample in nine parts of colorless *n*-heptane, as specified in Section 8(*d*).

Outline of Method

3. Using the north sky filter and 20-mm. cell, the photoelectric colorimeter is set to read 100 per cent transmittance with distilled water in the cell. The observed transmittance of the sample is determined and corrected to the north sky transmittance by means of predetermined calibration graphs. The north sky transmittance is reported as the color index of the sample for values from 3.0 to 95.0 per cent. For north sky trasmittances greater than 95.0 per cent, the measurement is repeated, using the violet filter, and the color index is calculated empirically. For north sky transmittances less than 3.0 per cent, the measurement is repeated with the north sky filter, using a solution of the sample in *n*-heptane, and the color index is obtained from an empirically derived table. When it is desired to obtain additional information concerning the color of the sample, the red transmittance is determined and used to characterize the color as normal or abnormal.

Apparatus

4. *Photoelectric Colorimeter, Sample Cells, Color Filters, and Glass Color Standards* as specified in the Supplement.

Diluent

5. *n-Heptane.*—Essentially pure *n*-heptane having a color index of 99.5 or higher.

Standardization of Apparatus

6. (*a*) Measure the transmittance values of the standards listed in Table I according to the procedure in Section 7, using distilled water in a 20-mm. cell as reference and using the appropriate color filters; use the same cell that is to be used for distilled water in Section 7, and place the standards in the cell compartment adjacent to the photocell. Obtain the difference between these observed values and the certified values furnished by the National Bureau of Standards, the certified values to be obtained in accord with the definitions given in Section A4 of the Supplement. Consider the apparatus unsuitable for use if the observed tramsmittances differ from the certified transmittances by more than the limits shown in

TABLE I.—COLORIMETER RESPONSE REQUIREMENTS.

Standard	Allowable Tolerances from N.B.S. Certified Percentage Transmittance Values		
	NORTH SKY FILTER	VIOLET FILTER	RED FILTER
N.B.S. No. 12..........	±3	±5	...
N.B.S. No. 13..........	±3	±5	...
N.B.S. No. 14..........	±5	±2	±3
N.B.S. No. 15..........	±5	...	±5
N.B.S. No. 16..........	±2	...	±4
N.B.S. No. 21..........	±5	±1	±5
N.B.S. No. 22..........	±5	±2	±1
Lampblack............	±0.5	±0.5	±0.5

Table I (Note 2). Repeat the standardization at monthly intervals, or more often if necessary, to guard against appreciable changes in instrument response.

NOTE 2.—An instrument can in some cases be adjusted to meet the requirements of this section by appropriate manipulation of the lamp rheostat or light reduction devices, or both.

(*b*) Considering standards Nos. 12 through 16 only, subtract the observed transmittances obtained relative to distilled water with the north sky filter from the corresponding certified value for the standard furnished by the National Bureau of Standards. Assign to the difference thus obtained the appropriate algebraic sign; thus, if the observed relative transmittance is smaller than the certified value, assign a positive sign to the difference, and, if the observed transmittance is greater than the certified value, assign a negative sign to the difference. Prepare a correction graph (Note 3) for the north sky filter by plotting the differences against the observed values and connecting each plotted point to its neighbor with a straight line; draw the graph through zero difference at 100 per cent transmittance and through the difference between zero and the observed value for lampblack at zero transmittance. Use the values taken off this graph, with their algebraic signs, as corrections to be applied to observed transmittance readings. Make similar graphs for the red and violet filters.

NOTE 3.—If all the transmittance differences for any filter obtained in accordance with Paragraph (*b*) are less than 0.5 unit, the appropriate correction graph need not be prepared.

Procedure

7. (*a*) Adjust the colorimeter in accordance with the manufacturer's operating instruction so that a galvanometer deflection of 10 to 50 millimeter scale divisions will result upon changing the slide wire dial from 100 to 90 per cent transmittance, using the north sky filter and having the instrument balanced at 100 per cent transmittance for distilled water in the sample cell.

(*b*) Adjust the temperature of the sample to 25 ± 5 C. (Note 4). Take the pair of matched 20-mm. sample cells (Note 5) one of which was used in the preparation of the correction graphs (Section 6). Fill one cell with distilled water and the other with sample; allow any entrained air bubbles to escape. Polish the outside surfaces of the cells with a clean, lint-free cloth or tissue, and place the water-filled cell in the colorimeter. Wipe the north sky filter with a clean lint-free cloth or tissue, put the filter in position in the colorimeter, and adjust the colorimeter to read 100 per cent transmittance. Replace the water cell with the cell containing the sample and measure the transmittance of the sample (Note 6), estimating the nearest 0.1 per cent. Reinsert the water cell and check the instrument adjustment. If this reading differs from 100 per cent by more than 0.5 per cent, readjust the colorimeter and obtain a new reading for the sample.

NOTE 4.—With proper precautions, satisfactory results may sometimes be obtained with samples that require heating to eliminate a cloud or to melt the sample. In such cases, the sample should not be heated over 5 C. above the cloud point or 15 C. above the melting point. In any event, the method should not be used for samples that must be heated above 40 C.

NOTE 5.—Clean the sample cells before use by immersion in chromic acid cleaning solution. Rinse thoroughly, first with distilled water and then with acetone, and dry with a stream of clean air. If a series of samples is being analyzed, it suffices to rinse out the cell with a hydrocarbon solvent and then with acetone and dry.

NOTE 6.—The light passing through the sample may cause a change in color of unstable products. With such samples, the measurements should be made as rapidly as possible to minimize this effect and the degree of this instability should be mentioned in the report.

(*c*) If the north sky transmittance value is above 95.0 per cent, obtain the violet transmittance for the sample, using the violet filter and adjusting the instrument to a galvanometer deflection of 10 to 50 millimeter scale divisions per 10 per cent transmittance units as directed in Paragraph (*a*), but with the violet filter in place of the north sky filter; otherwise proceeding as directed in Paragraph (*b*).

(*d*) If the north sky transmittance value is below 3.0 per cent, prepare a solution consisting of one part by weight of sample to nine parts by weight of *n*-heptane and obtain the north sky transmittance as directed in Paragraph (*b*).

TABLE II.—DETERMINATION OF COLOR INDEX[a] FROM NORTH SKY TRANSMITTANCE OF HEPTANE SOLUTION OF THE SAMPLE.

H[b]	0	1	2	3	4	5	6	7	8	9
0	0.00	0.003	0.005	0.008	0.010	0.013	0.015	0.018	0.020	0.023
10	0.025	0.028	0.030	0.033	0.035	0.038	0.040	0.043	0.045	0.048
20	0.050	0.053	0.055	0.058	0.060	0.063	0.065	0.068	0.70	0.073
30	0.075	0.078	0.080	0.083	0.085	0.088	0.090	0.093	0.095	0.098
40	0.10	0.12	0.15	0.18	0.21	0.25	0.30	0.35	0.41	0.47
50	0.55	0.64	0.74	0.86	0.98	1.1	1.3	1.5	1.7	1.9
60	2.2	2.5	2.8	3.1	3.5	4.0	...	...	...	...

[a] For heptane solutions having north sky transmittances below 40, the color index is taken as ($0.0025H$); above 40, the color index is based on the expression:

$$\text{Color index} = \text{antilog}\,[2\text{-}7.5(2\text{-}\log H)]$$

[b] The symbol H = north sky transmittance of the *n*-heptane solution of the sample, in per cent.

(*e*) It is possible that certain oils, either because of the unusual nature of their crude source or the fact that they have been subjected to some unconventional method of refining, will possess exceptional color properties. Accordingly, if further characterization of the color of the sample is desired, obtain the red transmittance for the sample, using the red filter and adjusting the instrument to a galvanometer deflection of 10 to 50 millimeter scale divisions per 10 per cent transmittance units as directed in Paragraph (*a*), but with the red filter in place of the north sky filter; otherwise proceeding as directed in Paragraph (*b*).

(*f*) All transmittance measurements shall be made using the same light intensity and color temperature (that is, the same applied voltage, lamp rheostat setting, light reduction device, etc.) as used in the standardization of the apparatus (Section 6).

Calculation and Report

8. (*a*) Determine the correction for the observed reading, obtained as directed in Section 7, from the appropriate correction graph prepared as directed in Section 6 (*b*). Apply this correction to the observed reading and obtain the north sky, red, or violet transmittance.

(*b*) If the north sky transmittance of the sample is between 3.0 and 95.0 per cent inclusive, report that number to the closest 0.1 unit as the color index.

(*c*) If the north sky transmittance of the sample is greater than 95.0 per cent, calculate the color index as follows:

$$\text{Color index} = 90 + 0.1\ V$$

where V = violet transmittance in per cent.

Report the number so obtained to the closest 0.1 unit as the color index.

(*d*) If the north sky transmittance of a 20-mm. layer of the sample is less than 3.0, determine the color index from Table II; report the closest number given in the table.

(*e*) If the red transmittance of the sample was obtained, apply this corrected value and the north sky transmittance to Fig. 2. (Note 7.) If these values give a point lying within the two curves, report the color as "normal"; if not, report as "abnormal." Do not attempt to classify a sample as "normal" or "abnormal" if the north sky transmittance of a 20-mm. layer is less than 3.0.

NOTE 7.—Figure 2 shows a graph whose abscissa is north sky transmittance and whose ordinate is red transmittance, formed by two curves passing through the following points:

North Sky Transmittance, per cent	Red Transmittance, per cent	
	Minimum	Maximum
0	0	0
5	14	30
10	27	47
20	47	67
30	62	78
40	72	85
50	80	90
60	85	94
70	89	96
80	92	98
90	95	99
100	97	100

These data were taken from the Proposed Method of Test for Color of Lubricating Oil by

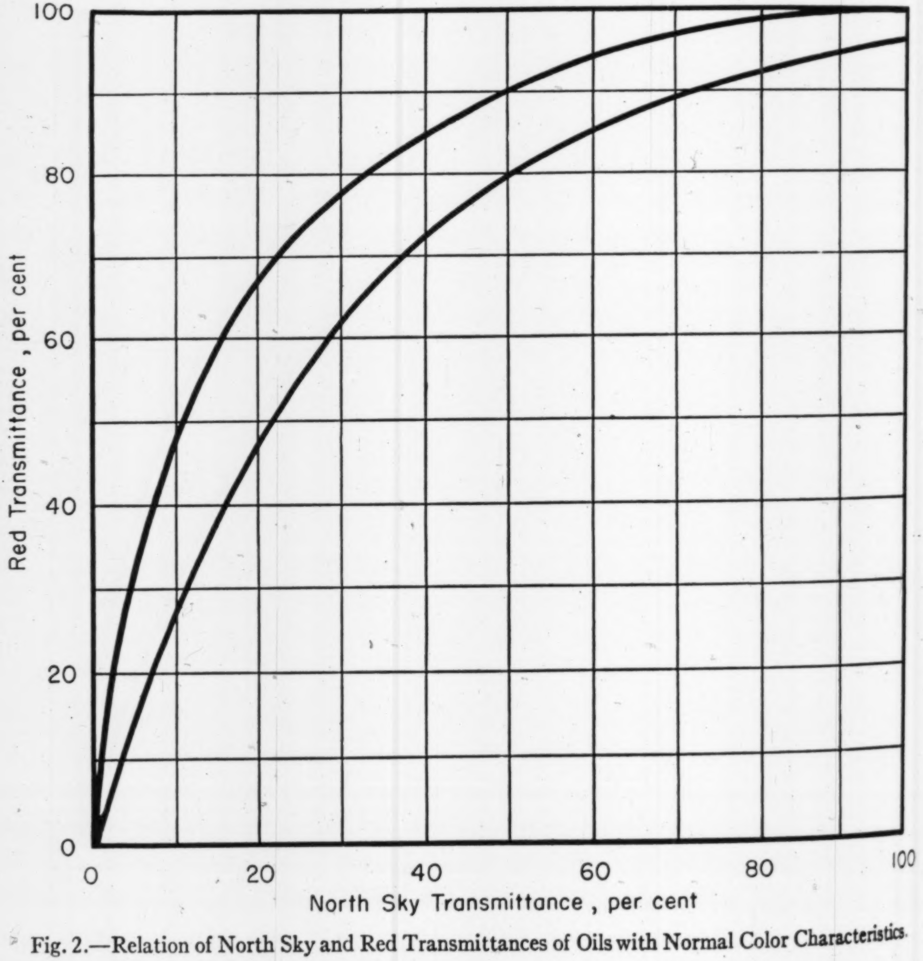

Fig. 2.—Relation of North Sky and Red Transmittances of Oils with Normal Color Characteristics.

Means of Photoelectric Colorimeter, 1942 Report of A.S.T.M. Committee D-2, Appendix II.[2] The data are based upon tests made on a large number of oils which are considered as being properly representative of those normally encountered in the petroleum industry. The term "abnormal" is arbitrarily assigned.

Precision

9. The results should not differ from the mean by more than the following amounts:

COLOR INDEX	Repeatability, One Operator and Apparatus	Reproducibility, Different Operators and Apparatus
0.0 to 2.9	0.2	0.4
3.0 to 95.0	1.0	2.0
95.1 to 100	0.5	1.0

NOTE 8.—Results on samples that are markedly "off-color" cannot in general be expected to show the precision given in Section 9.

SUPPLEMENT

APPARATUS

Photoelectric Colorimeter:

A1. The photoelectric colorimeter[3] shall conform to the response requirements prescribed in Section 6 and shall be made up of the following components:

(*a*) *Light Source.*—The light source shall conform to the following requirements:

(*1*) The light source shall be an incandescent tungsten lamp designed to operate at a color temperature of approximately 3000 K. The lamp base shall be so constructed that the lamp may be reproducibly positioned and may be centered with respect to the optical elements of the colorimeter.

(*2*) The colorimeter shall be equipped with a constant voltage source whose output is constant to ± 1 per cent.

(*3*) The light source circuit shall contain a rheostat or other device to permit adjustment of the color temperature of the lamp from approximately 2000 to 3000 K.

(*4*) The colorimeter shall be provided with suitable means to dissipate the heat generated by the light source; upon continuous operation for 1 hr. the temperature of the sample cell compartment shall not be more than 5 C. above room temperature.

(*b*) *Optical System.*—The optical system shall conform to the following requirements:

(*1*) The light from the lamp shall be passed through an optical system that will cause it to be incident substantially at right angles to the surface of the petroleum product.

(*2*) The filter holder shall reproducibly position the specified color filters.

(*3*) Means shall be provided for variably reducing the amount of light reaching both photocells without altering the spectral characteristics of the light. (Suitable means may consist of a series of reduction plates having holes of various diameters or metal screens having various ratios of opaque to transparent areas.)

(*4*) The sample cell and glass calibration standards described in Sections A2 and A4, respectively, shall be supported in such a manner that they are reproducibly positioned upon random insertion.

(*5*) The measuring and balance photocells shall be matched and of such type and response that the instrument will meet the requirements of Section 6. The adjustment of the colorimeter to read 100 per cent transmittance with distilled water in the sample cell shall be made by irradiation of the balance photocell.

(*c*) *Photoelectric Circuit.*—The photoelectric system may contain the following elements connected in a current bridge circuit:

(*1*) Measuring and balance photocells.

(*2*) A slide wire resistance equipped with a control dial graduated from 0 to 100 in units of 0.5 per cent transmittance, having linear response over the entire range.

(*3*) A galvanometer, having a coil resistance of 100 ± 20 ohms and a sensitivity of 0.06 ± 0.02 microamperes per millimeter scale division, used as a null indicator (the photoelectric circuit being adjusted until the galvanometer shows a deflection of zero scale divisions).

(*4*) A key switch that connects the galvanometer to the photoelectric circuit when held closed.

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[3] The Lumetron Model 402E Photoelectric Colorimeter, manufactured by the Photovolt Corp., 95 Madison Ave., New York 16, N. Y., has been found to be satisfactory for this purpose.

Sample Cells:

A2. Glass sample cells[4] shall have parallel planar sides and shall be of proper dimensions to permit use in the colorimeter employed. The length of the light path through the interior of the cell shall be 20.0 ± 0.1 mm.

TABLE III.—BASIC DATA DEFINING THE NORTH SKY, VIOLET, AND RED COLOR FILTERS.

WAVE LENGTH, mμ	RELATIVE SPECTRAL IRRADIANCE, COLOR TEMPERATURE 2,800 K., E	RELATIVE SPECTRAL RESPONSE OF PHOTOCELL, S	SPECTRAL TRANSMITTANCE OF THE PROTOTYPE FILTERS, RELATIVE TO WATER, PER CENT		
			NORTH SKY, T_{ns}	VIOLET, T_v	RED, T_r
350.....	4.33	0.30	1.6	49.0	0.0
360.....	5.32	0.34	10.0	60.0	0.0
370.....	7.26	0.38	21.0	70.0	0.0
380.....	9.12	0.42	30.0	74.0	0.0
390.....	11.30	0.47	34.0	76.0	0.0
400.....	13.82	0.51	40.8	73.8	0.0
410.....	16.71	0.56	41.8	68.5	0.0
420.....	19.94	0.60	44.0	59.4	0.0
430.....	23.54	0.65	45.8	49.2	0.0
440.....	27.52	0.69	47.4	39.3	0.0
450.....	31.86	0.73	49.9	29 2	0.0
460.....	36.55	0.76	52.0	18.8	0.0
470.....	41.00	0.80	53.8	9.3	0.0
480.....	47.03	0.84	55.5	3.1	0.0
490.....	52.71	0.87	56.4	0.4	0.0
500.....	58.78	0.90	56.8	0.1	0.0
510.....	65.08	0.93	56.8	0.0	0.0
520.....	71.64	0.95	56.2	0.0	0.0
530.....	78.45	0.97	54.8	0.0	0.0
540.....	85.45	0.98	53.0	0.0	0.0
550.....	92.68	0.99	50.8	0.0	0.0
560.....	100.0	0.98	48.7	0.0	0.0
570.....	107.4	0.96	46.2	0.0	0.0
580.....	115.1	0.94	42.9	0.0	0.0
590.....	122.7	0.90	39.3	0.0	0.0
600.....	130.4	0.84	35.4	0.0	7.0
610.....	138.1	0.77	31.5	0.0	28.0
620.....	145.8	0.65	27.5	0.0	40.4
630.....	153.5	0.53	23.4	0.0	42.2
640.....	161.1	0.40	19.9	0.0	40.8
650.....	168.6	0.26	16.4	0.0	38.4
660.....	176.1	0.15	13.5	0.0	36.0
670.....	183.2	0.09	10.8	0.0	33.5
680.....	190.5	0.05	8.5	0.0	31.3
690.....	197.5	0.03	6.4	0.0	29.8
700.....	204.4	0.02	4.7	1.0	28.2
710.....	211.3	0.014	3.3	3.2	26.9
720.....	217.7	0.010	2.2	5.0	25.7
730.....	224.0	0.006	1.3	5.8	24.8
740.....	230.2	0.004	0.7	6.1	24.1
750.....	236.1	0.003	0.3	6.1	23.5
760.....	241.7	0.0020	0.15	6.0	23.0
770.....	247.2	0.0015	0.08	5.8	22.5
780.....	252.4	0.0010	0.04	5.6	22.2
790.....	257.4	0.0007	0.02	5.5	22.0
800	262.2	0.0005	0.01	5.4	22.0

Color Filters:

A3. The light filters[5] shall be made of essentially plane glass, or combinations of glass, in which the coloring material is uniformly dispersed. The filters shall retain their color indefinitely. They shall be of a suitable size and shape to permit use in the colorimeter employed. Three filters designated as north sky, violet, and red shall be provided. The spectral characteristics of these filters shall be approximately as shown in Table III. These filters, in combination with the photoelectric colorimeter, shall meet the performance requirements prescribed in Section 6.

Glass Color Standards:

A4. The standards shall consist of seven essentially plane, 2-in. square, colored glasses or combinations of glasses. These standards shall be obtained from the National Bureau of Standards with certified north sky $(T/T_o)_{ns}$, violet $(T/T_o)_v$, and red $(T/T_o)_r$ trasmittances relative to distilledwater, defined as follows:

$$(T/T_0)_{ns} \equiv \sum_0^\infty ET_{ns}S(T/T_0)\Delta\lambda \Big/ \sum_0^\infty ET_{ns}S\Delta\lambda$$

$$(T/T_0)_v \equiv \sum_0^\infty ET_vS(T/T_0)\Delta\lambda \Big/ \sum_0^\infty ET_vS\Delta\lambda$$

$$(T/T_0)_r \equiv \sum_0^\infty ET_rS(T/T_0)\Delta\lambda \Big/ \sum_0^\infty ET_rS\Delta\lambda$$

where:

T/T_o = the spectral transmittance of the standard relative to a 20-mm. cell, with glass end plates, filled with distilled water,

E = the relative spectral irradiance of a source at a color temperature of 2800 K. (C_2 = 14,350) (see Table III),

S = the relative spectral response of the standard barrier-layer cell to a spectrum of unit irradiance given in Table III,

T_{ns}, T_v, and T_r = the spectral transmittances of the three prototypes of the North sky, violet, and red filters, respectively, given in Table III, and

$\Delta\lambda$ = the wave length interval, taken as 10 mμ.

These standards duplicate spectral characteristics of typical petroleum products.

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[4] Suitable cells, if not supplied with the colorimeter, may be obtained from the Klett Manufacturing Company, 177 E. 87th St., New York, N. Y.; from Fischer and Porter Company, 3507 Country Lane Road, Hatboro, Pa.; or from the Photovolt Corp., 95 Madison Ave., New York 16, N. Y.

[5] The Hellige-Diller 770-1 (north sky), 700-1 (red), and 760-2 (violet) filters supplied by Hellige, Inc., 3718 Northern Blvd., Long Island City, N. Y., are satisfactory.

APPENDIX V

PROPOSED METHOD OF TEST FOR SULFUR IN PETROLEUM PRODUCTS BY THE CO_2 - O_2 LAMP METHOD[1,2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method describes procedures for the determination of total sulfur in concentrations above 0.002 per cent by weight in liquid petroleum products. It is intended particularly for application to multiple analysis of two or more samples at the same time. The direct burning procedure (Section 6) is applicable to such materials as gasoline, kerosine, naphtha, and other liquids that can be burned completely and without smoking in a wick lamp. The blending procedure (Section 7) is applicable to aromatics, gas oils and distillate fuel oils, naphthenic acids, alkyl phenols, high sulfur content petroleum products, and many other materials that can not be burned satisfactorily by the direct burning procedure. Neither procedure is suitable for accurate analysis of samples that contain appreciable concentrations of halogens, phosphorus, nitrogen, or other acid-forming elements.

NOTE 1.—Results obtained by this method are expected to be identical with those obtained by A.S.T.M. Method D 90 - 47 T, Test for Sulfur in Petroleum Products by the Lamp - Gravimetric Method,[3] within the limits of precision and accuracy of the two methods. This method is appreciably more rapid than the lamp - gravimetric method when applied to routine multiple operations, and is generally to be preferred for such applications.

Outline of Method

2. The sample is burned in a closed system, using a wick-type lamp and an artificial atmosphere composed of 70 per cent carbon dioxide and 30 per cent oxygen to prevent formation of nitrogen oxides. The oxides of sulfur are absorbed and oxidized to sulfuric acid by means of hydrogen peroxide and, after flushing with air to remove dissolved carbon dioxide, the absorbent is titrated with sodium hydroxide solution.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Lamps, Chimneys, Absorbers, and Spray Traps* (Fig. 1) as specified in the Supplement

(*b*) *Cotton Wicking.*—Clean, unused, uniform, two-strand twisted cotton wicking of good quality, weighing 5 to 6 mg. per cm. per strand.

(*c*) *Manifold System* consisting of a vacuum manifold with regulating device, values, etc., and a dual manifold (burner and chimney) supplying a gas mixture of approximately 70 per cent CO_2 and 30 per cent O_2 at regulated pressures.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Published as information, October, 1947.

[3] 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

The vacuum manifold shall be connected to a pump of sufficient capacity to permit a steady gas flow about 3 liters per min. through each absorber, and to maintain a constant manifold pressure of approximately 40 cm. of water below atmospheric. The gas mixture in the burner manifold shall be maintained at a nearly constant pressure of 1 to 2 cm. of water. A suitable arrangement is described in the Supplement, but any other similar system may be used.

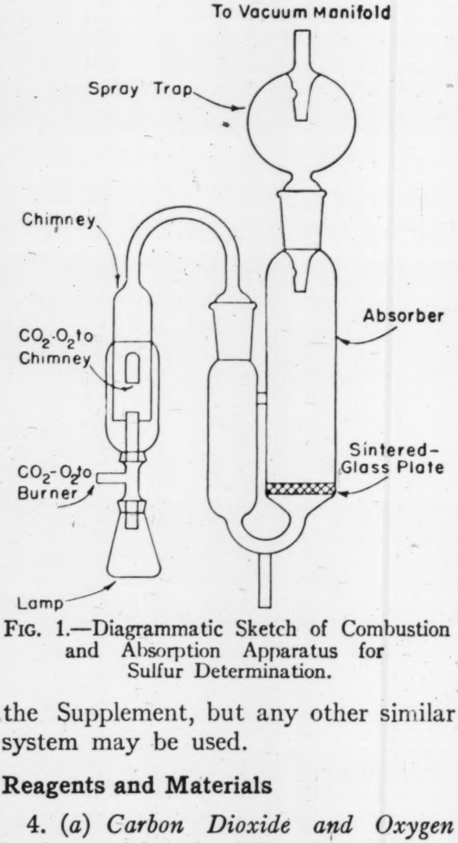

FIG. 1.—Diagrammatic Sketch of Combustion and Absorption Apparatus for Sulfur Determination.

Reagents and Materials

4. (*a*) *Carbon Dioxide and Oxygen* having purities of not less than 99.5 per cent, supplied in metal cylinders. These gases shall show no detectable sulfur by blank determination.

(*b*) *Diluent.*—The diluent used shall have a sulfur content less than 0.001 per cent, be completely miscible with the sample to be analyzed, and permit burning at a moderate rate without smoking. Normal heptane, *iso*octane, and absolute ethyl alcohol have been found suitable.

(*c*) *Hydrogen Peroxide* (*3 per cent*).—Dilute c.p. H_2O_2 (30 per cent) with distilled water. Store in a dark-colored glass-stoppered bottle.

(*d*) *Methyl Red Indicator Solution* (*0.1 per cent*).—Dissolve 0.1 g. of methyl red in 6 ml. of 0.05 *N* NaOH and dilute to 100 ml. with distilled water. Remove any undissolved solids by filtration.

(*e*) *Standard Sodium Hydroxide Solution* (*0.05 N*).—Dilute 2.8 ml. of saturated NaOH solution to 1 liter, using for this purpose the clear saturated solution decanted after standing long enough to permit any precipitate to settle out. Standardize by titration against standard acids in the absorber in the manner described in Section 8. Store in a chemically resistant glass bottle and protect to minimize contamination from CO_2 in the air.

NOTE 2.—The calculation of results may be simplified by adjusting the normality of the NaOH solution to 0.0624 ± 0.0001. One milliliter of the NaOH solution then will be equivalent to 0.0010 g. of sulfur.

Preparation of Apparatus

5. (*a*) When the apparatus is first assembled, adjust the individual valves between the vacuum manifold and the spray traps so that approximately 3 liters of air per minute will be drawn through each absorber when the chimney outlets are open to the atmosphere, the absorber is charged with 30 ± 2 ml. of water, and the pressure in the vacuum manifold is maintained at approximately 40 cm. of water below atmospheric. When all adjustments have been made, remove the water from the absorbers.

(*b*) Neutralize the H_2O_2 (3 per cent) immediately before use. As 30 ml. of the solution are needed, transfer to a beaker multiples of 30 ml. sufficient for the number of absorbers to be used simultaneously. Add one drop of methyl red indicator for each 100 ml. and add 0.05 *N* NaOH dropwise until the color changes from pink to yellow.

(*c*) Introduce 30 ± 2 ml. of the freshly neutralized H_2O_2 (3 per cent) into the larger bulb of each absorber. Attach the spray traps and chimneys and connect them to their respective manifolds by means of rubber tubing. Close the chimney openings by means of corks.

(*d*) With the burner control valves closed, the valve to the vacuum regulator fully open, and the pressure in the vacuum manifold adjusted to approximately 40 cm. of water below atmospheric, turn on the CO_2 and O_2. Adjust the chimney manifold control valve so that, at the required flow through the absorbers, only a small stream of gas escapes at the pressure regulator, a small stream of air enters at the vacuum regulator, and the pressure in the chimney manifold is 1 to 2 cm. of water. Minor adjustment of the vacuum regulator and vacuum control valve may be necessary to achieve this condition.

NOTE 3.—It is convenient to balance the gas flow system by regulating the pressure in the vacuum manifold. This is done by raising or lowering the air inlet tube in the vacuum regulator by sliding it in a rubber sleeve.

(*e*) Thread the required number of burners with four 15-cm. lengths of cotton wicking by doubling two 30-cm. lengths, inserting the looped ends into the top of the inner tube of the burner, and drawing the wicking through by means of a metal hook. Trim the wick as close as possible to the top of the burner with a pair of sharp scissors; then draw the wick down until the trimmed edge is flush with the top of the burner (see Note 5). It is essential that thoroughly cleaned burners and new wicking be used for each test.

Procedure for Direct Combustion

6. (*a*) By means of a pipet, introduce into the flask of each burner an approximate quantity of sample as indicated in Table I. Stopper the flasks with clean, numbered corks. Weigh each flask and its burner to 0.005 g.

NOTE 4.—While the stoppered flasks and prepared burners may all be weighed separately, it is usually more convenient to place each flask and its burner on the balance pan and obtain the combined weight in a single weighing.

(*b*) Handling each lamp individually, insert the burner in the flask. As soon as the sample has risen by capillary action to the top of the wick, connect the side tube of the burner to the burner manifold by means of rubber tubing. Light the burner with a sulfur-free flame (such as an alcohol lamp) and insert into the chimney, pinching off the connection between the chimney and the chimney manifold during the insertion if the flame tends to be blown out. At the same time, adjust the gas flow to the burner so that the flame is maintained at a point just below smoking and has a steady, symmetrical appearance. Continue in this manner until all lamps have been placed in the chimneys. Make any minor adjustment of the chimney control valve necessary to maintain the required pressure (see Section 5(*d*)). During the burning, and particularly during the latter stages when the flame becomes small, decrease the gas supply to the burners in order to prevent extinction of the flames.

TABLE I.

SULFUR CONTENT, per cent by weight	SAMPLE SIZE	
	g.	ml.
Under 0.05	10 to 15	20
0.05 to 0.3	5 to 10	10
0.3 to 1	3 to 5	5
Over 1	2 to 3	3

NOTE 5.—Most types of samples burn with a luminous yellow flame, the size and shape of which is dependent upon the gas flow to the burner, the volatility of the material, the tightness of the fit of the wick in the burner tube, and the position of the top of the wick relative to the top of the burner. It is preferable that the latter two variables be fixed with relation to the first before burning is started so that the flame can be controlled by variation in rate of gas flow. Highly volatile samples require a tight-fitting wick, the top of which may need to be several millimeters below the top of the burner, and in extreme cases may have to be cooled in ice during the burning. Less volatile materials require a more loosely fitting wick and require warming. In any case, it is essential that the flame burn smoothly and symmetrically and without jets in the inner cone or smoke on the outer fringes. Satisfactory combustion of materials difficult to burn can sometimes be obtained by increasing the O_2 content of the combustion atmosphere, but never above 40 per cent. Materials that can not be burned properly under those conditions shall be burned by blending as described in Section 7.

(*c*) When the burning of each sample is completed (as evidenced by the flame becoming small owing to depletion of the sample), remove the lamp from the chimney, extinguish the flame. shut off the CO_2 - O_2 supply to the burner and stopper the chimney opening. Immediately reweigh the lamp, burner, and numbered cork. When all combustions have been completed, turn off the CO_2 and O_2 supplies and close the connection to the vacuum regulator; this will cause air to be drawn into the chimney manifold through the manometer. Allow air to be drawn through the absorbers in this manner for 5 min. to remove dissolved CO_2 from the absorbent; then close the vacuum control valve.

NOTE 6.—If it is desired to conserve the combustion atmosphere, the gas flow through each individual absorber may be turned off upon completion of the burning period. To accomplish this, pinch off the rubber tubing connecting the spray trap to the vacuum manifold, reduce the flow of mixed gases at the rotameters proportionately, and readjust the vacuum control valve and the chimney control valve. When the burning of all samples has been completed, it is necessary to remove the pinch clamps and readjust the vacuum control valve in order to draw air at the required rate through the absorbers for removal of dissolved CO_2.

(*d*) Rinse the chimneys and spray traps with 30 to 40 ml. of distilled water, adding the rinsings to the absorbers, and proceed as directed in Section 8.

(*e*) *Blank.*—Leave the chimney of the blank absorber (Section 5(*c*)) stoppered, and allow the CO_2 - O_2 stream to pass through that absorber until all samples started at one time have finished burning. Titrate the absorber liquid as described below. Normally the combustion gas blank will be negligible, but if the blank titration is more than 0.1 ml. discard the determination and replace the CO_2 cylinder.

TABLE II.

SULFUR CONTENT, per cent by weight	SAMPLE SIZE	
	g.	ml.
0.5 and under	3 to 4	5
Over 0.5	2 to 3	3

Procedure for Blending and Combustion

7. (*a*) Add 6 ml. of sulfur-free diluent to each flask. Stopper the flasks with numbered corks and weigh to 0.005 g. By means of a pipet, introduce into the flask of each burner an approximate quantity of sample as indicated in Table II; swirl to mix thoroughly and reweigh (see Note 4).

NOTE 7.—Alternatively, make a quantitative 40 per cent blend of the sample in sulfur-free diluent and proceed as described in Section 6.

(*b*) Insert the burner and burn as described in Section 6 (*b*). Remove each lamp from its chimney as the flame nears extinction and extinguish the flame. Add 2 ml. of diluent, allowing the diluent to rinse down the walls of the flask.

Burn the additional diluent, and repeat the addition of diluent and burning one more time so that a total of 10 ml. of diluent has been burned.

(*c*) After all lamps have completed burning, turn off the CO_2 and O_2 supplies, close the connection to the vacuum regulator, draw air through the absorbers for 5 min., and finally close the vacuum control valve. Rinse the chimneys and spray traps with 30 to 40 ml. of distilled water, adding the rinsings to the absorbers, and proceed as directed in Section 8.

Analysis of Absorbent

8. Add 3 to 4 drops of methyl red indicator solution to the liquid in each absorber. Titrate the absorbent by introducing 0.05 *N* NaOH solution from a buret into the smaller bulb of the absorber. Use a 10 ml. microburet if less than 10 mg. of sulfur is expected to be present in the absorber. Stir during the titration by applying suction intermittently with the mouth to the top of the larger bulb (preferably by means of a short length of rubber tubing fitted to a perforated rubber stopper of appropriate size).

NOTE 8.—When incomplete combustion of the sample occurs, the absorber liquid will foam excessively. Also, during the titration, the air drawn through the absorber will have a characteristic taste or odor and the end point will be broad. In such cases, discard the determination.

Calculation

9. Calculate the sulfur content of the sample as follows:

$$\text{Sulfur, per cent by weight} = \frac{AN \times 1.603}{W}$$

or, if the normality of the NaOH solution has been adjusted to 0.0624 ± 0.0001:

$$\text{Sulfur, per cent by weight} = \frac{A}{W \times 10}$$

where:

A = milliliters of NaOH solution used in titrating the acid in the sample absorbent,

N = normality of the NaOH solution, and

W = grams of sample used.

NOTE 9.—When blends are used as described in Note 7, modify the calculations by using the measured aliquot portion of a sample, *W*.

Precision and Accuracy

10. (*a*) Results should not differ from the mean by more than the following amounts:

Sulfur Content, per cent by weight	**Repeatability,** One Operator and Apparatus	**Reproductability,** Different Operators and Apparatus
0.002 to 0.25......	0.001	0.002
Over 0.25.........	0.5 per cent of mean	1 per cent of mean

(*b*) Results should not differ from the true value by more than the following amounts:

SULFUR CONTENT, per cent by weight	ACCURACY
0.002 to 0.25..........	0.002
Over 0.25.............	1 per cent of true value

SUPPLEMENT

Apparatus:

A1. The apparatus shall consist of the following:

(*a*) *Lamp.*—A lamp of chemically resistant glass, consisting of a 25-ml. Erlenmeyer flask and a burner that conforms to the dimensions shown in Fig. 2. The burner consists of two concentric glass tubes, the external tube having an arm, provided with standard-taper glass joints for connection with the flask and the chimney. The upper ends of both burner tubes shall be polished and shall have plane surfaces that are in the same horizontal plane. The burner shall have a 1-mm. opening near its

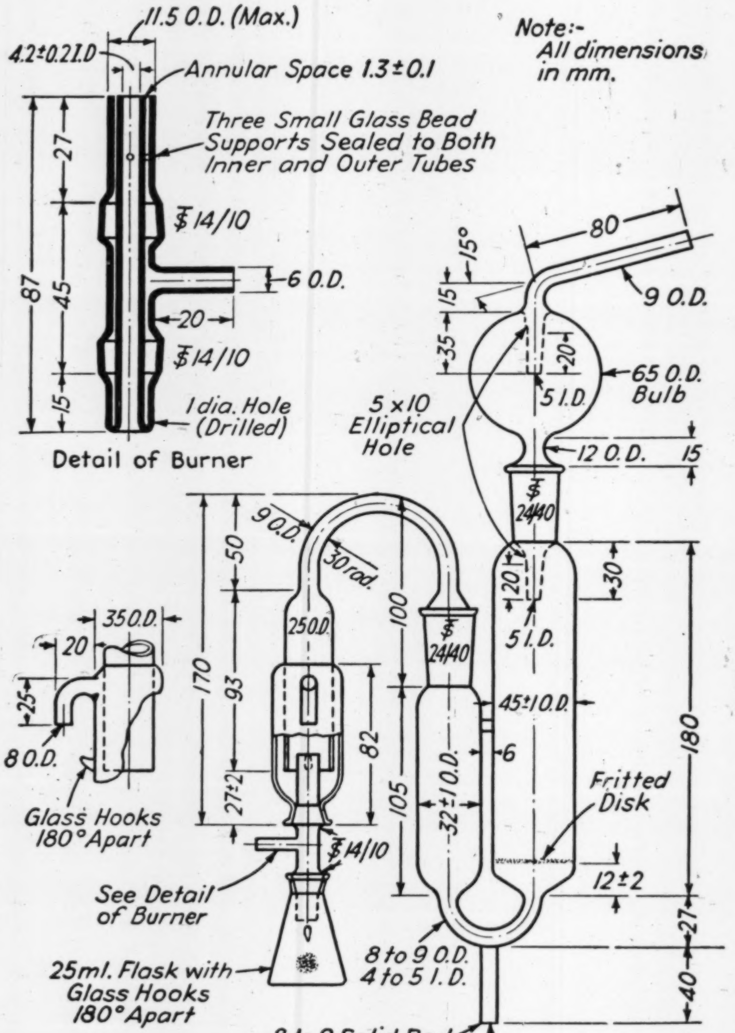
11.5 O.D. (Max.)
4.2±0.2 I.D.
Annular Space 1.3±0.1
Note:- All dimensions in mm.
Three Small Glass Bead Supports Sealed to Both Inner and Outer Tubes
27
87
45
15
$ 14/10
6 O.D.
20
$ 14/10
1 dia. Hole (Drilled)
Detail of Burner
80
15°
15
35
9 O.D.
20
5 I.D.
65 O.D. Bulb
5 x 10 Elliptical Hole
12 O.D.
15
$ 24/40
9 O.D.
30 rad.
50
170
100
30
20
5 I.D.
35 O.D.
20
25
8 O.D.
25 O.D.
93
$ 24/40
45±1 O.D.
6
82
180
Glass Hooks 180° Apart
27±2
105
32±1 O.D.
Fritted Disk
$ 14/10
12±2
See Detail of Burner
27
8 to 9 O.D.
4 to 5 I.D.
25 ml. Flask with Glass Hooks 180° Apart
40
8 to 9 Solid Rod
Held in Position by Any Suitable Support

FIG. 2.—Detailed Drawing of Combustion and Absorption Apparatus for Sulfur Determination.

NOTE.—In the case of those dimensions for which no specific tolerances are designated above, the permissible variation is plus or minus 10 per cent to the nearest millimeter, provided, however, that in no case shall the deviation be greater than 5 mm.

base to allow equalization of pressure between the chimney and the flask. When connected with the chimney, the lamp shall be held in position by rubber bands or metal springs stretched between glass hooks on the flask and chimney.

(*b*) *Chimney.*—A chimney of chemically resistant glass conforming to the dimensions shown in Fig. 2, provided with standard-taper glass joints for connection with the burner and absorber.

(*c*) *Absorber.*—An absorber of chemically resistant glass conforming to the dimensions shown in Fig. 2, provided with standard-taper glass joints for connection with the chimney and spray trap. A fritted disk with average pore diameter from 150 to 200 microns shall be sealed in the larger of two bulbs of the absorber. The fritted disk should be of such a porosity that, when 50 ml. of water is placed in the absorber and air is passed through at the rate of 3.0 liters per minute in the forward direction, the pressure differential between the two sides of the absorber is between 15 and 23 cm. of water and the air is dispersed uniformly.

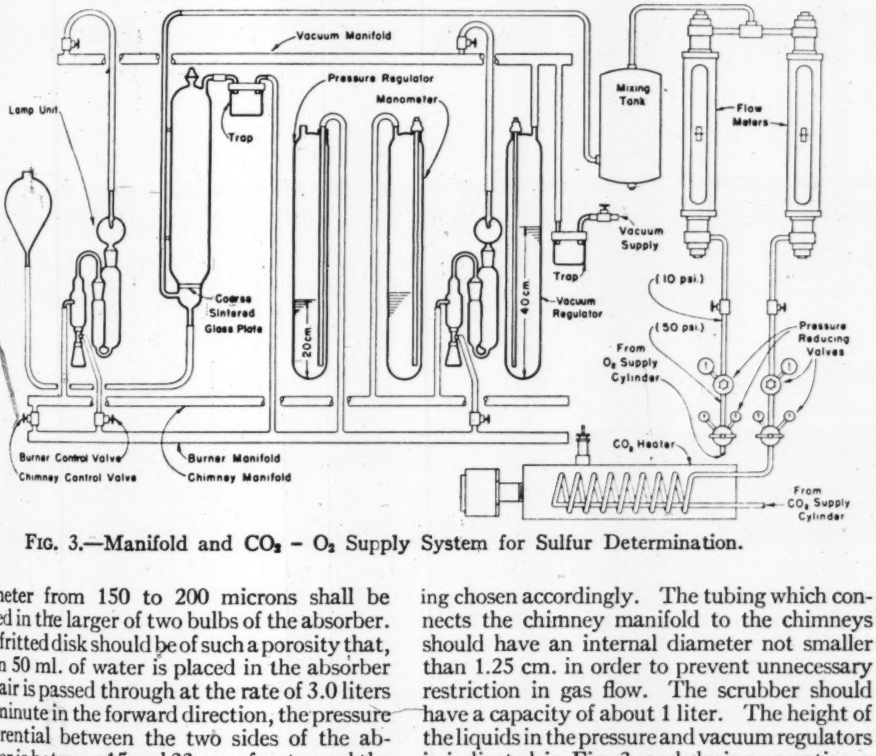
Vacuum Manifold
Pressure Regulator
Manometer
Lamp Unit
Trap
Coarse Sintered Glass Plate
20cm
40cm
Mixing Tank
Flow Meters
Vacuum Supply
Trap
Vacuum Regulator
(10 psi.)
(50 psi.)
From O_2 Supply Cylinder
Pressure Reducing Valves
CO_2 Heater
Burner Control Valve
Chimney Control Valve
Burner Manifold
Chimney Manifold
From CO_2 Supply Cylinder

FIG. 3.—Manifold and CO_2 - O_2 Supply System for Sulfur Determination.

(*d*) *Spray Trap.*—A spray trap of chemically resistant glass conforming to the dimensions shown in Fig. 2, provided with a standard-taper glass joint for connection with the absorber.

(*e*) *Manifold System.*—A satisfactory vacuum and combustion atmosphere manifold and supply system for supplying the required CO_2 - O_2 mixture to the lamp assemblies is shown diagrammatically in Fig. 3. The gases are supplied from commercial cylinders, the pressure of each gas being adjusted to 10±2 psi. by means of two single-stage regulating valves to ensure constant pressure at the flow regulating needle valves. It is necessary to pass the CO_2 through a heat exchanger installed ahead of the regulating valves to prevent freezing of the valves. The gases are passed through a metering system consisting of two calibrated rotameter flow meters to indicate the proportion of the two gases mixed in the surge tank. As many as eleven lamp assemblies can be operated as a unit, the throughput of the flow meters being chosen accordingly. The tubing which connects the chimney manifold to the chimneys should have an internal diameter not smaller than 1.25 cm. in order to prevent unnecessary restriction in gas flow. The scrubber should have a capacity of about 1 liter. The height of the liquids in the pressure and vacuum regulators is indicated in Fig. 3, and during operation a slow leak of gas through them should be maintained.

NOTE 10.—In use, place 300 to 400 ml. of H_2O_2 (3 per cent) in the scrubber. Since the manifold manometer also serves as a scrubber, use H_2O_2 (3 per cent) as the manometric liquid. Replace weekly or whenever the volume becomes less than two thirds of the original.

APPENDIX VI

PROPOSED RESEARCH PROCEDURE FOR DETERMINATION OF MINERAL OIL CONTENT OF USED LUBRICATING OIL[1]

This is a proposed procedure and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Subcommittee X on Insoluble Content of Used Oils, of Committee D-2 on Petroleum Products and Lubricants, in connection with its research investigation of the changes in a lubricating oil which take place in service, has developed a procedure for the separation of mineral oil from substantially all contaminating matter and additives in a used oil. The committee does not have data correlating the results of this procedure with service data. Sufficient information[2] has been submitted to justify recommending the procedure as a research analytical method for the determination of the mineral oil content of a used lubricating oil. It is requested that research laboratories using this procedure submit information both on the use of the procedure and on the correlation of results with service performance to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method describes a research procedure for determining the mineral lubricating oil content of used lubricating oils. It is applicable only to used oils containing more than 2 per cent of contaminating matter and addition agents from any lubricating oil system operating under conditions that promote oxidation of the oil, including oils from aircraft, automotive, and stationary internal combustion engines and from laboratory oxidation apparatus.

NOTE 1.—In addition to the matter insoluble in *n*-pentane removed from used oils by A.S.T.M. Method D 893, Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils,[3] this procedure also removes contaminating matter, additives soluble in *n*-pentane, and volatile matter such as water and diluent from fuel.

Definitions

2. (*a*) *Used Lubricating Oil* is the oil and suspended contaminating matter which circulates through a lubricating oil system during operation and which can be drawn from the oil system during or immediately following a period of operation. It does not include deposits in the system or large contaminating particles that may have separated from the oil in the system.

(*b*) *Mineral Lubricating Oil Content* of a used lubricating oil is that portion remaining after substantially complete removal of contaminating matter and addition agents. The contaminating matter may include dirt, carbon, products of combustion, products of the oxidation and thermal decomposition of the oil, coolants, and diluents.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, October, 1947.

[2] "Oxidation of Petroleum Lubricants," L. L. Davis, Bert H. Lincoln, G. D. Byrkit, and W. A. Jones, *Industrial and Engineering Chemistry*, Vol. 33, March, 1941, p. 339.

"The Examination of Used Engine Crankcase Oil," L. L. Davis, ASTM BULLETIN, Vol. 113, December, 1941, p. 13.

[3] 1946 Book of A.S.T.M. Standards, Part III-A.

Outline of Method

3. The sample of lubricating oil is dissolved in a heavy naphtha, the contaminating matter and addition agents are adsorbed with a decolorizing clay, and the mineral lubricating oil content is determined from an aliquot of the clear supernatant solution.

Apparatus

4. (*a*) *Evaporation Bath*, constructed as described in A.S.T.M. Method D 381, Test for Existent Gum in Gasoline (Air-Jet Evaporation Method),[3] but changed for evaporations at 100 ± 3 C. with an air flow of 60 ± 5 liters per min. The bath shall also meet the performance test described in Note 2.

NOTE 2.—Weigh to the nearest 1 mg., into an evaporation beaker, 2.5 ± 0.1 g. of new, straight mineral, S.A.E. 30 grade, lubricating oil. Add 40 ml. of Stoddard solvent (Section 5 (*b*)), stir with a glass rod, and wash the rod with 5 ml. of solvent. Evaporate at the prescribed temperature and air rate for 60 ± 5 min., cool, and weigh the remaining oil to the nearest 1 mg. The average change in weight of the oil for five determinations shall be not greater than 0.2 per cent. If necessary, adjust the time of evaporation to meet the required performance.

(*b*) *Evaporation Beakers.*—Berzelius-type beakers of heat-resistant glass, without lip, of 100-ml. nominal capacity.

(*c*) *Centrifuge*, electric, capable of whirling the filled centrifuge tubes at a speed not less than that calculated by the following formula:

$$\text{Revolutions per minute} = \frac{6000}{\sqrt{D}}$$

where D = the diameter, in inches, of swing (tip to tip of whirling tubes) of the centrifuge tubes.

(*d*) *Centrifuge Tubes*, having a gross capacity of 250 ml.

Materials

5. (*a*) *Decolorizing Clay.*—Fuller's earth[4] of Georgia-Florida quality; at least 98 per cent passing a No. 200 sieve.

(*b*) *Stoddard Solvent*, conforming to A.S.T.M. Specifications D 484, for Stoddard Solvent.[3] The residue obtained by evaporating 50 ml. of solvent, as directed in Section 7 (*d*), shall not exceed 2 mg.

Preparation of Sample

6. Heat the sample of used oil to 60 ± 5 C. in the original container and agitate until all sediment is homogeneously suspended in the oil (Note 3). If the original container is of opaque material or if it is more than three-fourths full, transfer the entire sample to a clear glass bottle having a capacity at least one-third greater than the volume of the sample and transfer all traces of sediment from the original container to the bottle by violent agitation of portions of the sample in the original container. After complete suspension of all sediment, strain the sample or a convenient aliquot through a 100-mesh screen for the removal of large contaminating particles.

NOTE 3.—As used oil may change appreciably in storage, samples should be tested as soon as possible after removal from the lubricating system and the dates of samplng and testing should be noted.

Procedure

7. (*a*) *Preparation of Evaporation Bath.*—Adjust the air rate to 60 ± 5 liters per min. Heat the bath and adjust the temperature controller so that the temperature of the effluent air, measured

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[4] Suitable fuller's earth may be obtained from the Attapulgus Clay Co., 260 S. Broad St., Philadelphia 2, Pa.; or from the Floridin Company, Warren, Pa.

by a thermometer with the bulb resting on the bottom of a beaker at the center, is 100 ± 3 C. When the air temperature is under control, read the temperature of a thermometer placed in the bath well. Thereafter, during the evaporation, the well temperature shall not vary by more than 3 C. Heat two clean evaporation beakers in the bath at 100 ± 3 C. for at least 1 hr., cool in a covered vessel containing no drying agent, and weigh to the nearest 1 mg.

(*b*) *Evaporation Correction Blank.*—Weigh to the nearest 1 mg., into the first evaporation beaker, 2.5 ± 0.1 g. of the prepared sample. Add about 40 ml. of Stoddard solvent from 50 ml. in a pipet, stir with a glass rod until the oil is in solution, wash the oil from the rod into the beaker with the remaining 10 ml. of solvent, and set aside in a clean place.

(*c*) *Adsorption of Contaminating Matter and Additives.*—Weigh to the nearest 10 mg., into a clean 250 ml. centrifuge tube, 10 ± 0.1 g. of the prepared sample. Record the weight of the oil and that of the tube plus oil. Add 200 ml. of Stoddard solvent and weigh to the nearest 0.1 g. Stopper and shake until the oil is in solution. Add 2 ± 0.25 g. of decolorizing clay, stopper, and shake vigorously for 100 vertical 12-in. strokes; a mechanical shaker giving an equivalent degree of agitation may be used. Centrifuge until the supernatant solution is clear. If the supernatant solution has a reddish-yellow hue, add another 2 g. increment of clay, shake, and centrifuge (Note 4). Continue adding 2-g. increments of clay, shaking, and centrifuging until the hue changes from amber (reddish yellow) to yellow. The contaminating oxidized oil adds a reddish hue to the predominantly yellow hue of the mineral lubricating oil, and the final removal of such contaminating matter is indicated by the change in hue with an insignificant change in color density (see Supplement). Centrifuge until the supernatant solution is clear and free of suspended clay. Pipet 50 ml. of clear solution from the centrifuge tube into the second dried and weighed evaporation beaker, and weigh to the nearest 0.1 g.

NOTE 4.—With highly oxidized oils, larger than 2-g. increments of clay may be used until the color end point is approached, after which 2-g. increments should be used. At least two increments immediately preceding the end point shall be 2 ± 0.25 g. For convenience in measuring the clay, a dipper may be used having such volume that it delivers 2 ± 0.25 g. of a given sample of clay.

(*d*) *Evaporation.*—Remove the conical adapters from two of the wells of the evaporation bath as prepared in Paragraph (*a*), place the beakers containing the decolorized oil and blank solutions in the wells, and replace the adapters. Evaporate the solvent for 60 ± 5 min. Remove the beakers, cool in a covered vessel under the same conditions used in Paragraph (*a*) for at least 1 hr., and weigh to the nearest 1 mg. If there is a gain in weight of the blank of more than 2 mg., indicating incomplete evaporation of the naphtha, repeat the determination and, if necessary, adjust the conditions of evaporation in accordance with Note 2.

Calculation and Report

8. (*a*) Calculate the evaporation correction, D, for the blank as follows:

$$D = \frac{(A - B)C}{A} - E$$

where:

A = weight of sample used in blank evaporation test, in grams,

B = weight of sample after evaporating the blank, in grams,

C = weight of sample used for analysis, in grams, and

E = weight of water contained in the sample C, in grams, (as determined by A.S.T.M. Method D 95, Test for Water in Petroleum Products and other Bituminous Materials).[3]

Note 5.—An evaporation correction of 2 mg. or less is considered of no significance and may be disregarded.

(*b*) Calculate the aliquot fraction used, I, as follows:

$$I = \frac{F - G}{H + D}$$

where:

F = weight of 50-ml. aliquot of solution, in grams,

G = weight of oil after evaporation of solvent from aliquot, in grams, and

H = weight of 200 ml. of solvent used, in grams.

(*c*) Calculate the mineral lubricating oil content, J, of the sample in per cent by weight as follows, and report to the nearest 0.1 per cent:

$$J = \frac{G}{CI} \times 100$$

Precision

9. Results should not differ from the mean by more than the following amounts:

Mineral Lubricating Oil Content, per cent	Repeatability, One Operator and Apparatus	Reproducibility, Different Operator and Apparatus
Over 90..........	0.4	0.7
75 to 90..........	0.6	1.0
Under 75..........	0.8	1.5

SUPPLEMENT

Determination of the Color End Point in the Adsorption of Contaminating Matter

A1. (*a*) The accurate determination of the mineral lubricating oil content of a used oil depends upon the recognition of the color change occurring when all oxidation products have just been removed by the clay from the solution. During the adsorption step, the first increments of clay remove insoluble matter, additives, and asphaltenes, leaving the solution a dark red to reddish amber color, dependent upon the amount of oxidation products in the oil. The final increments of clay remove the last trace of oxidation products and cause a change in hue from amber (reddish yellow) to yellow. The color end point is indicated by this change in hue rather than change in the color density. Additional increments of clay continue to lighten the depth of color but make no appreciable change in yellow hue.

(*b*) An inexperienced operator may have difficulty in recognizing the color end point. It is recommended that skill be developed by several trial experiments, using different quantities of clay, so that the end point is well bracketed. Visual examination of the centrifuged solutions will permit the selection of the quantity of clay which just caused the change in hue. The procedures described in Sections A2 and A3 are suggested for the determination of the color end point.

A2. *Colorimetric Measurement.*—Proceed with the adsorption of the contaminating matter as directed in Section 7 (*c*) until the clear solution is reddish amber in color. Pipet a portion of the clear solution into a colorimeter cell and measure the color density, using a green filter (540 mμ). The measurement may be in any units proportional to concentration. Return the solution from the cell to the centrifuge tube, add another increment of clay, centrifuge, and again measure the color, using the same cell. Repeat until the end point has been passed by at least two increments of clay. Plot the color densities against grams of clay used. Two straight lines may be drawn through the points representing the colors before and after the end point. The intersection of these lines indicates the true end point. Repeat the determination, using the amount of clay indicated by the intersection.

A3. *Visual Comparison.*—If a colorimeter is not available, the end point may be determined by retaining a small sample of solution in a test tube for comparison with the solution after the succeeding clay increment. The color is observed in flat-bottomed glass test tubes about 12½ mm. in diameter, using "daylight" reflected from a white matte surface. Proceed as directed in Section A2 until the end point is approached; then pipet sufficient clear solution into a test tube to give a depth of 40 ± 1 mm. Add an increment of clay to the centrifuge tube, shake, and centrifuge. Pipet the clear solution into a second test tube and compare the two solutions, looking through the tubes. If the second solution shows a markedly lighter color than the first but still retains appreciable reddish hue, return the solution from the first (darker) test tube to the centrifuge tube and add another increment of clay. Repeat until the new solution shows little difference in density of color but does show a yellowish rather than amber tint as compared to the previous sample. With the end point thus reached, proceed with the measurement and evaporation of the aliquot portion of the solution.

APPENDIX VII

PROPOSED METHOD OF TEST FOR ACID AND BASE NUMBERS OF PETROLEUM OILS BY ALKALI BLUE COLOR INDICATOR[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. (*a*) This method is intended for determination of acidic or basic constituents (Note 1) in petroleum products and lubricants and for other materials soluble or nearly soluble in mixtures of benzene and isopropyl alcohol. It is applicable for the determination of acids or bases whose dissociation constants in water are larger than 10^{-9}; extremely weak acids or bases whose dissociation constants are smaller than 10^{-9} do not interfere. Salts react if their hydrolysis constants are larger than 10^{-9}.

NOTE 1.—In new and used oils, the constituents considered to have acidic characteristics include organic and inorganic acids, esters, phenolic compounds, lactones, resins, salts of heavy metals, and addition agents such as inhibitors and detergents. Similarly, constituents considered to have basic properties include organic and inorganic bases, amino compounds, salts of weak acids (soaps), basic salts of polyacidic bases, salts of heavy metals, and addition agents such as inhibitors and detergents.

(*b*) This method may be used to indicate relative changes that occur in an oil during use under oxidizing conditions. Although the titration is made under definite equilibrium conditions, the method does not measure an absolute acidic or basic property that can be used to predict performance of an oil under service conditions. No general relationship between bearing corrosion and acid or base numbers is known.

NOTE 2.—Oils, such as many cutting oils, rustproofing oils, and similar compounded oils, or excessively dark-colored oils, which cannot be analyzed by this method due to insolubility in the titration solvent or obscurity of the color-indicator end point, can be analyzed by A.S.T.M. Method D 664, Test for Acid and Base Numbers of Petroleum Products by Electrometric Titration.[2] The acid or base numbers obtained by this color-indicator method may or may not be numerically the same as those obtained by Method D 664, but they are generally of the same order of magnitude. Results obtained by the former A.S.T.M. Method D 188 and by A.S.T.M. Method D 663, Test for Acid and Base Numbers of Petroleum Oils by Color-Indicator Titration,[2] are not comparable with those obtained by this color-indicator method.

Definitions

2. (*a*) *Total Acid Number.*—The quantity of base, expressed in milligrams of potassium hydroxide, that is required to titrate all acidic constituents present in 1 g. of sample.

(*b*) *Strong Acid Number.*—The quantity of base, expressed in milligrams of potassium hydroxide, that is required to titrate the strong acid constituents present in 1 g. of sample.

(*c*) *Strong Base Number.*—The quantity of acid, expressed in terms of the equivalent number of milligrams of potassium hydroxide, that is required to titrate the strong base constituents present in 1 g. of sample.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, October, 1947.

[2] 1946 Book of A.S.T.M. Standards, Part III-A.

Outline of Method

3. To determine the total acid or strong base number, the sample is dissolved in a neutralized titration solvent (benzene and isopropyl alcohol containing a small amount of water) to which the alkali blue indicator has been added, and the resulting single-phase solution is titrated at room temperature with standard alcoholic base or alcoholic acid solution, respectively, to the end point indicated by the color change of the added alkali blue solution (purple-blue in acid and brick-red in base). To determine the strong acid number, a separate portion of the sample is extracted with hot water and the aqueous extract is titrated with potassium hydroxide solution, using methyl orange as indicator.

Apparatus

4. *Buret.*—A 50-ml. buret graduated in 0.1-ml. subdivisions, or a 10-ml. buret graduated in 0.05-ml. subdivisions.

Reagents

5. (*a*) *Standard Potassium Hydroxide Solution* (*alcoholic, 0.1 N*).—Add 6 g. of c. p. solid KOH to approximately 1 liter of c. p. anhydrous isopropyl alcohol (containing less than 0.9 per cent water) in a 2-liter Erlenmeyer flask. Boil the mixture gently for 10 to 15 min., stirring to prevent the solids from forming a cake on the bottom. Add at least 2 g. of c. p. $Ba(OH)_2$ and again boil gently for 5 to 10 min. Cool to room temperature, allow to stand for several hours, and filter the supernatant liquid through a fine sintered-glass or porcelain filtering funnel; avoid unnecessary exposure to CO_2 during filtration. Store the solution in a chemically resistant dispensing bottle out of contact with cork, rubber, or saponifiable stopcock lubricant and protected by a guard tube containing soda lime or soda asbestos (Ascarite). Standardize frequently enough to detect changes of 0.0005 *N* (Note 3), preferably against pure potassium acid phthalate in about 100 ml. of carbon dioxide-free water, using phenolphthalein to detect the end point.

NOTE 3.—To simplify calculations, both the standard KOH and HCl solutions may be adjusted so that 1.00 ml. is equivalent to 5.00 mg. of KOH. Sodium hydroxide and sulfuric acid may be substituted for potassium hydroxide and hydrochloric acid, respectively.

(*b*) *Standard Hydrochloric Acid Solution* (*alcoholic, 0.1 N*).—Mix 9 ml. of c. p. HCl (sp. gr. 1.18) with 1000 ml. of c. p. anhydrous isopropyl alcohol (containing less than 0.9 per cent water). Standardize frequently enough to detect normality changes of 0.0005 (Note 3), preferably by electrometric titration of approximately 8 ml. (accurately measured) of the standard 0.1 *N* alcoholic KOH solution diluted with 125 ml. of carbon dioxide-free distilled water.

(*c*) *Titration Solvent.*—Add 500 ml. of c. p. benzene and 5 ml. of water to 495 ml. of c. p. anhydrous isopropyl alcohol.

(*d*) *Alkali Blue Indicator.*[3]—Extract 4 g. of solid alkali blue, in a Soxhlet apparatus equipped with a paper thimble, with ethyl alcohol (95 per cent) for 6 hr. Cool the solution, filter if necessary, and dilute with ethyl alcohol (95 per cent) to 200 ml., or to a volume that will contain at least 0.5 g. of dissolved alkali blue per 100 ml. Store the indicator solution in a chemically resistant bottle protected from strong light.

NOTE 4.—Solutions prepared and sold as indicators may be used for nonreferee determinations. As a precaution, such solutions should not be used without trial, as their strength is often insufficient to meet the specifications.

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[3] Alkali blue (solid indicator grade known as alkali blue 6B) is available from the following sources:
Allied Chemical and Dye Corp., National Aniline Div., 40 Rector Street, New York 6, N. Y.
Coleman and Bell Co., Manufacturing Chemists, Norwood, Ohio.
Eimer & Amend, Third Avenue, Eighteenth to Nineteenth Sts., New York, N. Y.

(*e*) *Methyl Orange Indicator Solution.*—Dissolve 0.1 g. of methyl orange in 100 ml. of distilled water.

Preparation of Sample

6. (*a*) Strict observance of the sampling procedure described in Paragraph (*b*) is necessary, since the sediment itself is acidic or basic or has adsorbed acidic or basic material from the sample. Failure to obtain a representative sample causes serious errors.

(*b*) Heat the sample of used oil to 60 ± 5 C. in the original container and agitate until all sediment is homogeneously suspended in the oil (Note 5). If the original container is of opaque material, or if it is more than three-fourths full, transfer the entire sample to a clear glass bottle having a capacity at least one-third greater than the volume of the sample, and transfer all traces of sediment from the original container to the bottle by violent agitation of portions of the sample in the original container. After complete suspension of all sediment, strain the sample or a convenient aliquot through a 100-mesh screen for the removal of large contaminating particles.

NOTE 5.—As used oil may change appreciably in storage, samples should be tested as soon as possible after removal from the lubricating system and the dates of sampling and testing should be noted.

Procedure for Total Acid Number

7. (*a*) To 100 ml. of the titration solvent, add 3 ml. of the alkali blue indicator solution and a drop of 0.1 *N* HCl to sensitize the indicator. Neutralize the solution with 0.1 *N* KOH to a strong wine-red color. Do not stop at the preliminary purple-red color.

NOTE 6.—The indicator must be activated as described in Section 7(*a*) before every titration.

(*b*) Into a 250-ml. Erlenmeyer flask, introduce a weighed quantity of the sample as given in Table I. Add 100 ml. of the neutralized titration solvent containing freshly activated indicator and, without stoppering, swirl until the sample is entirely dissolved by the solvent. If the mixture assumes a purple-blue color, proceed as directed in Paragraph (*c*); if it becomes a brick-red color, proceed as directed in Section 8.

(*c*) Titrate immediately at a temperature below 30 C. Add 0.1 *N* KOH in increments and swirl to disperse the KOH as necessary. Shake vigorously near the end point, but avoid dissolving CO_2 in the solvent. (In the case of acidic oils, the blue color changes to a purple-blue as the end point is approached.) Consider the end point reached when the purple-blue color changes to a pronounced brick or claret-red color; consider the end point definite if the color change persists for 15 sec. or if it reverses with 2 drops of 0.1 *N* HCl.

TABLE I.—SIZE OF SAMPLE.[a]

Total Acid Number or Total Base Number	Size of Sample, g.	Sensitivity of Weighing, g.
NEW OR LIGHT-COLORED OILS		
0.0 to 3.0	20.0 ± 2.0	0.05
3.0 to 25.0	2.0 ± 0.2	0.01
25.0 to 250.0	0.2 ± 0.02	0.001
USED OR DARK-COLORED OILS		
0.0 to 25.0	2.0 ± 0.2	0.01
25.0 to 250.0	0.2 ± 0.02	0.001

[a] Light-colored samples of low acid number permit the use of 20-g. samples to obtain more precise results. The sample size for dark colored oils is limited to the quantity specified to minimize possible interference by the dark color.

NOTE 7.—To observe the end-point of dark-colored oil, shake the flask vigorously to produce momentarily a slight foam when the color change occurs as the last few drops of titrant are added and observe the titration under a white fluorescent lamp at bench-top level.

Procedure for Strong Base Number

8. If the titration solvent assumes a

brick-red color when added to the sample (Section 7(*b*)), carry out the titration as described in Section 7, but use 0.1 *N* HCl and titrate until the color changes from brick-red to purple-blue.

Procedure for Strong Acid Number

9. (*a*) Introduce approximately 25 g. of a representative sample, weighed to the nearest 0.1 g., into a 250-ml. separatory funnel and add 100 ml. of boiling distilled water. Shake vigorously and drain the water phase, after separation, into a 500-ml. casserole. Extract the sample twice more with 50-ml. portions of boiling distilled water, adding both extracts to the casserole. To the combined extracts add 0.1 ml. of methyl orange indicator solution and, if the solution becomes pink or red, titrate with 0.1 *N* KOH until the solution becomes golden brown in color. If the initial color is not pink or red, report the strong acid number as zero.

(*b*) *Blank.*—Make a blank determination on 100 ml. of the same boiling distilled water as used in the titration of the sample. Titrate with 0.1 *N* KOH to the same depth and shade of color obtained in the titration of the sample.

Calculations

10. (*a*) Calculate the total acid number and strong acid number as follows:

Total acid number, mg. KOH per g. =

$$\frac{AN \times 56.1}{W}$$

Strong acid number, mg. KOH per g. =

$$\frac{(B - C)N \times 56.1}{W}$$

where:

A = milliliters of KOH solution required for titration of the sample (Section 7),

N = normality of the KOH solution,

W = grams of sample used,

B = milliliters of KOH solution required for titration of the water extract (Section 9(*a*)), and

C = milliliters of KOH solution required for titration of the distilled water blank (Section 9(*b*)).

(*b*) Calculate the strong base number as follows:

Strong base number, mg. KOH per g. =

$$\frac{Dn \times 56.1}{W}$$

where:

D = milliliters of HCl required for titration of the sample (Section 8),

n = normality of HCl, and

W = grams of sample used.

Precision

11. Results should not differ from the mean by more than the following amounts:

Total Acid Number or Strong Base Number	**Repeatability,** One Operator and Apparatus	**Reproducibility,** Different Operators and Apparatus
NEW OR LIGHT COLORED OILS		
0.0 to 0.5	0.05	0.1
0.5 to 3.0	0.1	0.2
3.0 to 25.0	0.3	0.6
25.0 to 250.0	2.0	4.0
USED OR DARK COLORED OILS		
0.0 to 0.5	0.2	0.4
0.5 to 25.0	0.5	1.0
25.0 to 250.0	2.0	4.0

NOTE 8.—These precision values do not apply to oils that are partially immiscible in the solvent or that are so highly colored as to obscure the end-point color change.

APPENDIX VIII

PROPOSED METHOD OF TEST FOR ACID AND BASE NUMBERS OF PETROLEUM OILS BY ALPHA-NAPHTHOLBENZEIN COLOR INDICATOR[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. (Same as Section 1 of the Proposed Test for Acid and Base Numbers of Petroleum Oils by Alkali Blue Color Indicator.)[2]

NOTES 1 and 2.—(Same as Notes 1 and 2 of the Alkali Blue Method.)

Definitions

2. (Same as Section 2 of the Alkali Blue Method.)

Outline of Method

3. To determine the total acid or strong base number, the sample is dissolved in a mixture of benzene and isopropyl alcohol containing a small amount of water, and the resulting single-phase solution is titrated at room temperature with standard alcoholic base or alcoholic acid solution, respectively, to the end point indicated by the color change of the added alpha-naphtholbenzein solution (orange in acid and green-brown in base). To determine the strong acid number, a separate portion of the sample is extracted with hot water and the aqueous extract is titrated with potassium hydroxide solution, using methyl orange as an indicator.

Apparatus

4. (Same as Section 4 of the Alkali Blue Method.)

Reagents

5. (*a*) (Same as Section 5(*a*) of the Alkali Blue Method.)

NOTE 3.—(Same as Note 3 of the Alkali Blue Method.)

(*b*) (Same as Section 5(*b*) of the Alkali Blue Method.)

(*c*) (Same as Section 5(*c*) of the Alkali Blue Method.

(*d*) *Alpha-Naphtholbenzein Indicator Solution.*—Prepare a solution containing 10 g. of alpha-naphtholbenzein[3] per liter of c. p. anhydrous isopropyl alcohol. The solid alpha-naphtholbenzein shall contain less than 0.5 per cent chlorine and shall be in the form of a chocolate-brown amorphous powder.

(*e*) *Methyl Orange Indicator Solution.*—Dissolve 0.1 g. of methyl orange in 100 ml. of distilled water.

Preparation of Sample

6. (Same as Section 6 of the Alkali Blue Method.)

NOTE 4.—(Same as Note 5 of the Alkali Blue Method.)

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, October, 1947.

[2] See p. 327.

[3] Solid alpha-naphtholbenzein is available from the Allied Chemical and Dye Corp., National Aniline Div., 40 Rector St. New York 6, N. Y.

Procedure for Total Acid Number

7. (*a*) Into a 250-ml. Erlenmeyer flask, introduce a weighed quantity of the sample as given in Table I. Add 100 ml. of the titration solvent and 3 ml. of the indicator solution, and, without stoppering, swirl until the sample is entirely dissolved by the solvent. If the mixture assumes a yellow-orange color, proceed as directed in Paragraph (*b*); if it becomes green or green-black, proceed as directed in Section 8.

Table I.—Size of Sample (Same as Table I of the Alkali Blue Method).

(*b*) Titrate immediately at a temperature below 30 C. Add 0.1 *N* KOH in increments and swirl to disperse the KOH as necessary. Shake vigorously near the end point, but avoid dissolving CO_2 in the solvent. (In the case of acidic oils, the orange color changes to a green or green-brown as the end point is approached.) Consider the end point definite if the color change persists for 15 sec. or if it reverses with 2 drops of 0.1 *N* HCl.

NOTE 5.—(Same as Note 7 of the Alkali Blue Method.)

(*c*) *Blank*.—Make a blank titration on 100 ml. of the titration solvent and 3 ml. of the indicator solution, adding 0.1 *N* KOH in 0.05- or 0.1-ml. increments. Record the quantity of 0.1 *N* KOH required to reach the end point (orange to green).

Procedure for Strong Base Number

8. If the titration solvent containing the dissolved sample assumes a green or greenish-brown color after the indicator is added (Section 7(*a*)), carry out the titration as described in Section 7, but use 0.1 *N* HCl and titrate until the green-brown color changes to orange.

Procedure for Strong Acid Number

9. (Same as Section 9 of the Alkali Blue Method.)

Calculations

10. (*a*) Calculate the total acid number and strong acid number as follows:

Total acid number, mg. KOH per g. =

$$\frac{(A - B)N \times 56.1}{W}$$

Strong acid number, mg. KOH per g. =

$$\frac{(C - D)N \times 56.1}{W}$$

where:

A = milliliters of KOH solution required for titration of the sample (Section 7(*b*)),

B = milliliters of KOH solution required for titration of the blank (Section 7(*c*)),

N = normality of the KOH solution,

W = grams of sample used,

C = milliliters of KOH solution required for titration of the water extract (Section 9(*a*)), and

D = milliliters of KOH solution required for titration of the distilled water blank (Section 9(*b*)).

(*b*) Calculate the strong base number as follows:

Strong base number, mg. KOH per g. =

$$\frac{(E - F)n \times 561}{W}$$

where:

E = milliliters of HCl solution required for titration of the sample (Section 8),

F = milliliters of HCl required for titration of the blank,

n = normality of the HCl solution, and

W = grams of sample used.

Precision

11. (Same as Section 11 of the Alkali Blue Method.)

NOTE 6.—(Same as Note 8 of the Alkali Blue Method.)

APPENDIX IX

PROPOSED METHOD OF TEST FOR TOTAL OLEFINIC AND AROMATIC HYDROCARBONS IN GASOLINE[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test is intended for the determination of olefinic plus aromatic hydrocarbons in gasolines or other hydrocarbon mixtures that have 95 per cent distillation temperatures of 205 C. (400 F.) or lower by A.S.T.M. Method D 86, Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products.[2] It is applicable to finished motor and aviation fuels, to base and blending stocks, and to any narrow-boiling hydrocarbon mixture falling within this boiling range limitation. The method is not applicable to determinations above 98.5 per cent olefins plus aromatics if a 10-ml. sample is used nor above 97.0 per cent if a 5-ml. sample is used (Note 1).

NOTE 1.—The ASTM has no correlation between this method and the similar test (aromatic hydrocarbon content of Stoddard Solvent) described in A.S.T.M. Specifications D 484, for Stoddard Solvent.[2]

Outline of Method

2. A measured volume of sample is shaken, at the temperature of melting ice, with a solution of phosphorus pentoxide in concentrated sulfuric acid. The contraction in volume due to the absorption of olefinic and aromatic hydrocarbons in the acid mixture is a measure of the total content of those constituents (Note 2).

NOTE 2.—The contraction in volume actually measures total olefins and aromatics present in the sample plus any volume change which occurred when these olefins and aromatics were mixed with the saturated hydrocarbons. The maximum volume change known among hydrocarbons of this boiling range is 0.6 per cent expansion for a 2:3 mixture of benzene and saturates. However, the volume change is generally less than 0.3 per cent; hence it is usually less than the precision of the method and may be neglected.

Apparatus

3. (*a*) *Standard and Precision Sulfonation Flasks* meeting the requirements shown in Figs. 1, 2, and 3.

(*b*) *Pipets* of 5 and 10-ml. capacities, calibrated to deliver 5.0 ± 0.02 ml. and 10.0 ± 0.04 ml. of water at 20 C., respectively.

(*c*) *Centrifuge*, mechanical or hand-driven, capable of a speed of 1000 rpm., and equipped with metal cups having a minimum inside diameter of $1\frac{1}{2}$ in. The construction of the centrifuge shall be such that no part of the neck of the flask touches the centrifuge head when the centrifuge is in motion. (Note 3.)

NOTE 3.—The standard two-cup, hand-driven, milk-test centrifuge for 6-in. Babcock bottles may be used when the standard sulfonation flasks (Fig. 1) are being centrifuged. The penny heads of the stoppers must be aligned in the same vertical plane to prevent breakage.

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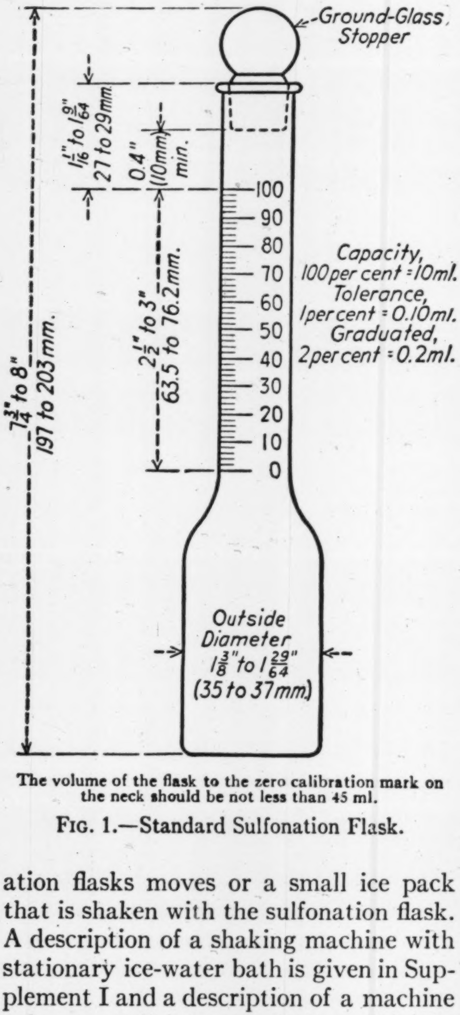
[1] This proposed method is under the jurisdiction of the A. S. T. M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1945; revised, 1946, 1947.

[2] 1946 Book of A.S.T.M. Standards, Part III-A.

(*d*) *Mechanical Shaker* having a horizontal stroke 3 ± 0.5 in. in length and a speed of 250 ± 25 cycles per min., each cycle consisting of one forward and one return stroke. The ice-water bath may be either a large stationary bath through which the carrier for the sulfonation flasks moves or a small ice pack that is shaken with the sulfonation flask. A description of a shaking machine with stationary ice-water bath is given in Supplement I and a description of a machine using a small ice pack shaken with the flask is given in Supplement II. The shaker shall shake the desired number of sulfonation flasks, rigidly clamped in an upright position or at an angle of 45 deg. to the direction of motion, while they are immersed to the 20 ± 10 per cent (or 2 ± 1 ml.) mark in a stationary ice bath. In a small ice pack shaken with the flask, the flask may be immersed to a greater depth.


Ground-Glass Stopper
1 1/16" to 1 9/64" 27 to 29 mm.
0.4" (10 mm) min.
100
90
80
70
60
50
40
30
20
10
0
Capacity, 100 per cent = 10 ml. Tolerance, 1 per cent = 0.10 ml. Graduated, 2 per cent = 0.2 ml.
2 1/2" to 3" 63.5 to 76.2 mm.
7 3/4" to 8" 197 to 203 mm.
Outside Diameter 1 3/8" to 1 29/64" (35 to 37 mm)

The volume of the flask to the zero calibration mark on the neck should be not less than 45 ml.

FIG. 1.—Standard Sulfonation Flask.

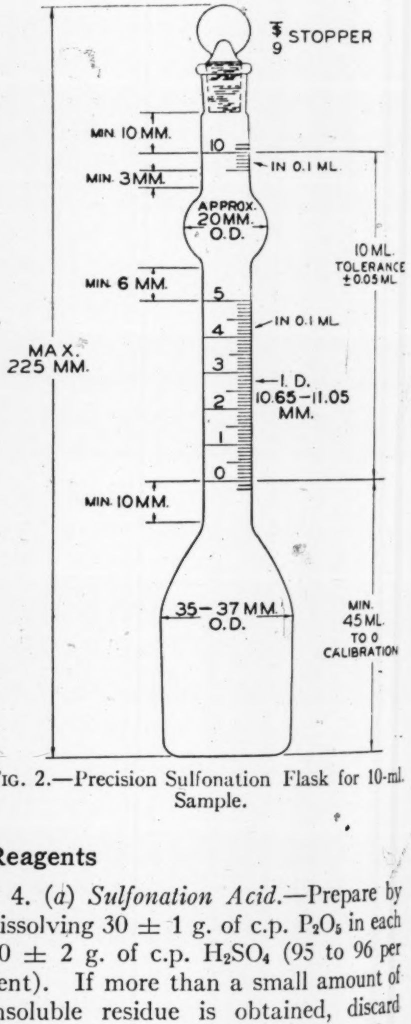

₸ 9 STOPPER
MIN. 10 MM.
MIN. 3 MM.
10
IN 0.1 ML.
APPROX. 20 MM. O.D.
10 ML. TOLERANCE ±0.05 ML.
MIN. 6 MM.
5
4
3
2
1
0
IN 0.1 ML.
MAX. 225 MM.
I. D. 10.65–11.05 MM.
MIN. 10 MM.
35–37 MM. O.D.
MIN. 45 ML. TO 0 CALIBRATION

FIG. 2.—Precision Sulfonation Flask for 10-ml. Sample.

Reagents

4. (*a*) *Sulfonation Acid.*—Prepare by dissolving 30 ± 1 g. of c.p. P_2O_5 in each 70 ± 2 g. of c.p. H_2SO_4 (95 to 96 per cent). If more than a small amount of insoluble residue is obtained, discard the reagent. Store in a 500-ml. glass stoppered "ether bottle." In storage, keep the ground-glass cap in place to prevent the collection of moisture on the lip of the bottle.

(*b*) *Sulfuric Acid, c.p.* (*95 to 66 per cent by weight*).

Test of Procedure Using Check Samples

5. (*a*) *Non-Aromatic Sample.*—Percolate 200 ml. of methyl-cyclohexane through 50 g. of silica gel packed tightly in a 10-mm. diameter column. Following the procedure given in Section 6, the total absorption (or apparent aromatic content) for this sample should not exceed 1 per cent (Note 4).

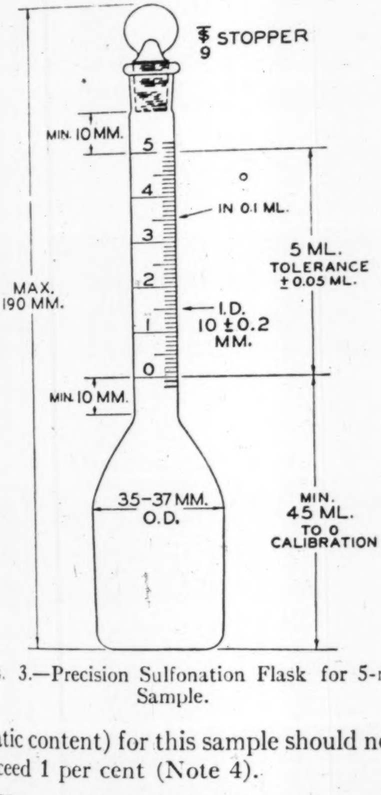


FIG. 3.—Precision Sulfonation Flask for 5-m Sample.

NOTE 4.—Significant departures from the specified procedure, particularly in regard to the reaction temperature, may yield results that differ from the correct value by as much as 5 per cent. Methylcyclohexane reacts more readily with the acid at temperatures above 0 C. than do paraffins or other naphthenes.

(*b*) *Aromatic Sample.*—Purify a paraffin (*iso*octane, *n*-heptane, etc.) by percolating a 200-ml. portion through 50 g. of silica gel in a 10-mm. column. Place 60 ± 0.1 ml. in a 100-ml. volumetric flask and dilute to volume with pure toluene.[3] Following the procedure given in Section 6, the aromatic content of this sample should be 40 ± 1 per cent and the raffinate should have a refractive index or density within plus or minus 0.0002 of that of the purified paraffin or naphthene used for the blend (Note 5).

NOTE 5.—Sufficient contact between the acid and hydrocarbon is a critical factor affecting the precision of the method, particularly when the toluene content is about 40 per cent.

Procedure

6. (*a*) Introduces 25 ± 1 ml. of the sulfonation acid into either the standard or precision sulfonation flask (depending on the desired precision) and close the flask with the glass stopper. Place the flask in its holder in the shaking machine so that it is submerged in the ice-water bath (0 to 4 C.) as described in Section 3 (*d*), and allow it to remain in the bath for at least 5 min. Measure and record the temperature of the sample. (*Caution,* Note 6.)

NOTE 6: *Caution.*—At this point the operator should put on a face mask or safety goggles.

(*b*) By means of a pipet, add 10 ml. of sample to the sulfonation acid, allowing the sample to run slowly down the walls of the flask so that no mixing of the two phases will occur (Note 7). Stopper the flask and secure in place in the shaking machine by means of the clamp (Note 8).

NOTE 7.—A highly volatile sample, such as a C_5, must be cooled in an ice-water bath and transferred to the sulfonation flask by means of a cooled pipet.

NOTE 8: *Caution.*—Excessive pressure, which may develop in blends of high olefin content, can cause breakage if the stopper is fastened rigidly to the flask. Therefore it is important that the stopper be fastened in such a way that it can move slightly and thus release the pressure. Either a sponge rubber disk placed between the stopper of the flask and the clamp, or a spring tension clamp, is satisfactory.

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[3] Eastman X grade toluene has been found satisfactory for this purpose.

(*c*) Allow the flask to stand for 5 min. after introduction of the sample into the flask; then cause the shaking machine to make two or three cycles (a cycle consists of a forward and a return stroke) at the end of each 10 sec. for the next 5 min. Then, without further interruption, allow the flask to be shaken at a rate of 250 ± 25 cycles per minute for the next 20 min. for vertical shaking, or for the next 15 min. for oblique shaking. (Note 9).

NOTE 9: *Danger.*—Put on face mask or goggles before proceeding with the next step in the procedure.

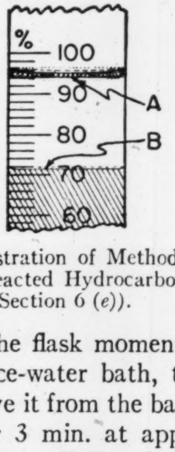

FIG. 4.—Illustration of Method for Reading Volume of Unreacted Hydrocarbon in Sulfonation Flask (see Section 6 (*e*)).

(*d*) Vent the flask momentarily while still in the ice-water bath, then immediately remove it from the bath and centrifuge it for 3 min. at approximately 1000 rpm. Pour H_2SO_4 (95 to 96 per cent) down the side of the neck of the flask until the hydrocarbon layer is entirely within the graduated portion of the neck, then centrifuge for an additional 5 min.

(*e*) Bring the acid-hydrocarbon mixture to a temperature within 1 C. (Note 10) of that at which the sample was measured by immersing the sulfonation flask for 5 min. in a beaker of water maintained at the desired temperature. Place the flask in front of a plain, nonglary, white or light-colored background illuminated by a diffused light. Read the scale to the nearest quarter division (that is, to the nearest 0.05 ml.) on a standard flask, and to the nearest 0.2 division (that is, to the nearest 0.02 ml.) on a precision flask, at the upper and lower levels of the hydrocarbon layer as represented by points *A* and *B* in Fig. 4. Point *A* denotes the nearly flat boundary between the clear hydrocarbon and the darkest part of the air-liquid interface, and point *B* refers to the lower and more distinct interface between the clear hydrocarbon and the sulfonation acid. (In Fig. 4 these readings are, respectively, 9.4 and 7.15 ml.)

NOTE 10.—A temperature change of 1 C. corresponds to a volume change of about 0.1 per cent.

(*f*) If the residual volume is less than 5 ml. and olefins are known to be present in the sample, make a second determination using only 5 ml. of sample. For such samples, the prescribed centrifuging may not be sufficient to completely separate all unreacted hydrocarbons from the acid layer. Therefore, the flask shall be centrifuged after the volume reading and the volume read again. If the second volume reading differs from the first reading, the centrifuging shall be repeated until two successive volume readings agree within 0.02 ml.

Calculation and Report

7. (*a*) Calculate the olefin-aromatic content as follows:

Olefin-aromatic content,

$$\text{per cent by volume} = \frac{(V - r)100}{V} - \frac{10C}{V}$$

where:

V = milliliters of sample,
r = milliliters of residue, and
C = correction for solubility of residue in the acid (see Table I).

TABLE I.—SOLUBILITY CORRECTIONS (SEE SECTION 7 (*a*)).

$(V - r)100/V$	C
0 to 5	0.7
5 to 20	0.8
20 to 35	0.9
35 to 45	1.0
45 to 60	1.1
60 to 70	1.2
70 to 85	1.3
85 to 100	1.4

(*b*) When results are obtained with both 5 and 10-ml. samples and the former are not more than 1.5 per cent different from the latter, report the values obtained with the 10-ml. sample; otherwise report the results obtained with the 5-ml. sample.

Precision

8. Results should not differ from the mean by more than the following amounts:

Type of Flask	Sample Size, ml.	Repeatability, One Operator and Apparatus	Reproducibility, Different Operators and Apparatus
Standard	5	2	3
Standard	10	1	2
Precision	5	1	2
Precision	10	0.5	1

(See Supplements, pp. 338 to 341)

SUPPLEMENT I

SHAKING MACHINE WITH STATIONARY ICE-WATER BATH

The stationary ice bath, as shown in Fig. 5, is mounted from the base of the shaking machine. It consists of a wooden box with an inner metal lining. The metal tank is 16 in. in length, 18 in. in depth, and has two partitions of $\frac{1}{4}$-in. mesh wire screen which divide the bath into three compartments each 6 in. in width. Ice is packed in the two outer compartments and the tank filled with water so that the sulfonation flasks are immersed to a depth of $3\frac{1}{2}$ in. The center compartment, in which the carrier for the sulfonation flasks moves, will then be filled with water free from ice at a temperature of between 0 and 2 C.

The shaking apparatus, with the ice bath removed to illustrate the mounting of the flask carrier on the reciprocating part of the machine, is shown in Fig. 6. The carrier assembly shown holds eight flasks, supported in pairs by clamps which screw down firmly over the tops of the flasks as shown in Fig. 7. The clamp consists of two inverted cups, each containing a sponge rubber disk, mounted on a plate. The cups cover the stoppers and upper necks of the flasks and serve as relief valves, should excessive pressures develop in the flasks during shaking.

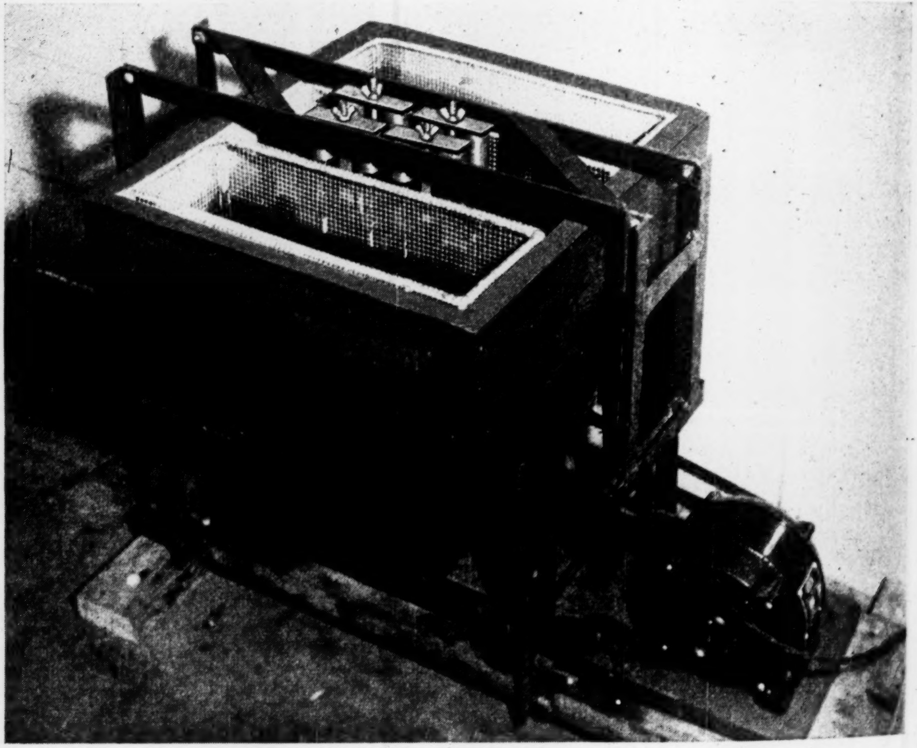

FIG 5.—Shaking Machine with Stationary Ice-Water Bath.

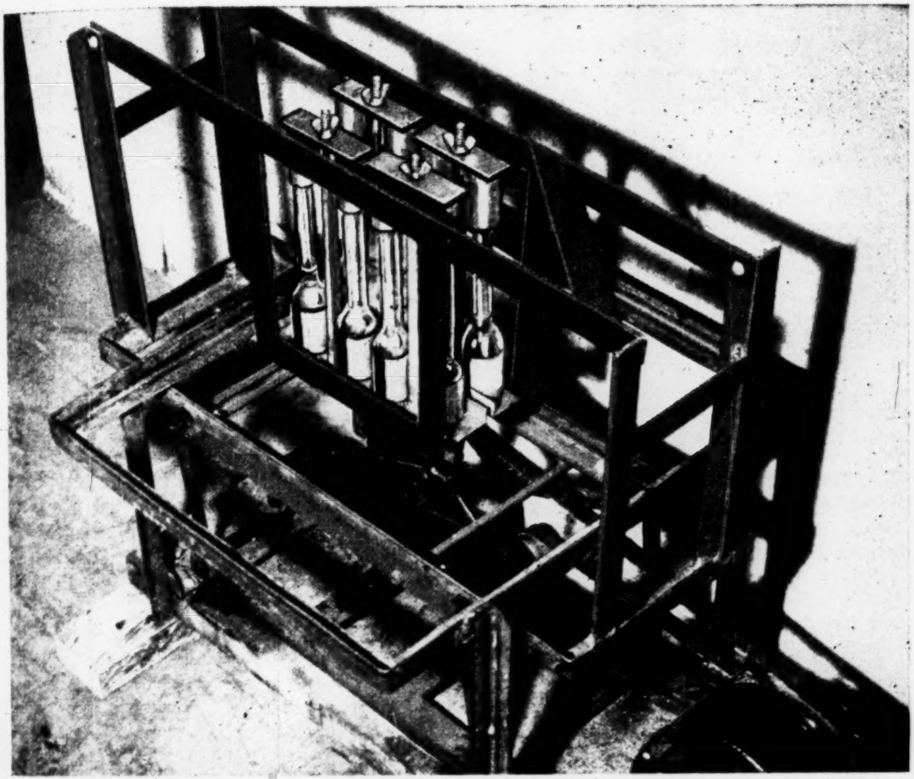

FIG. 6.—Shaking Machine with Ice Water Bath Removed.

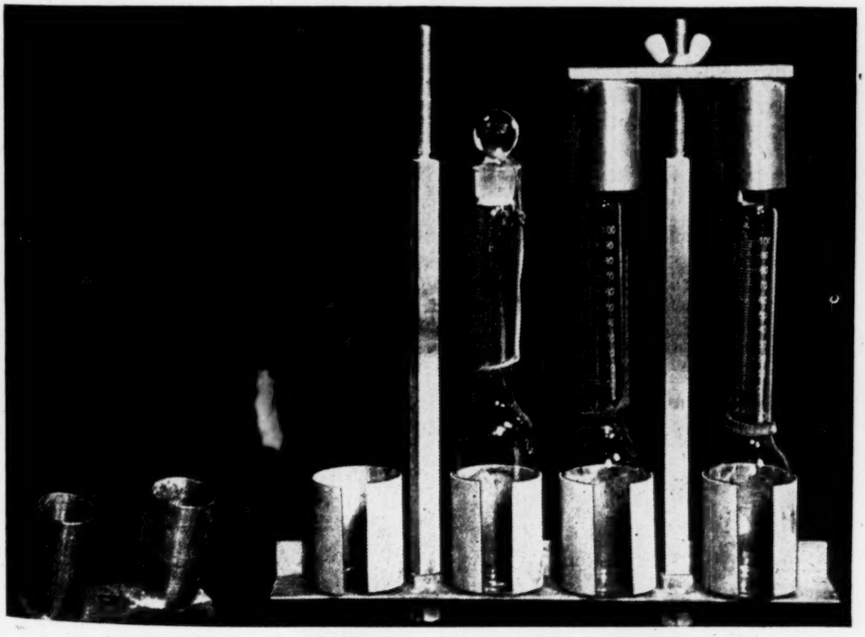

FIG. 7.—Sulfonation Flask Holder and Clamp for Shaking Machine.

SUPPLEMENT II

SHAKING MACHINE FOR INDIVIDUAL ICE-WATER BATH

Figure 8 shows a shaking machine with a carriage fitted with a wooden block and clamp arrangement for holding four cooling jars rigidly at an angle of 45 deg. and pointing in the direction of motion. These cooling jars provide a means for shaking sulfonation flasks which are immersed in an ice-water slurry. A suitable jar may be made by drilling a hole of appropriate size through the plastic lid of a 500-ml. capacity chemical reagent jar, as shown in Fig. 9. The neck of the flask extends through this opening and is cushioned by a short piece of rubber tubing. The stopper of the flask is held in place by a modified Sligh oxidation flask clamp, the head of the stopper being ground if necessary to permit use of this clamp.

The stoppered sulfonation flask containing the acid is placed in the empty cooling jar which is then packed with finely crushed ice, keeping the flask properly centered. The jar is completely filled with water to displace all the air, and the lid screwed on the jar. After the sample is pipetted into the flask, the stopper is clamped in position. It is important that spring tension of the modified Sligh clamp (Fig. 8) be adjusted so as to allow a slight movement of the stopper of the sulfonation flask under pressures that may develop when shaking highly olefinic samples. The apparatus is covered with a cloth during shaking to catch any spray released if excessive pressures develop.

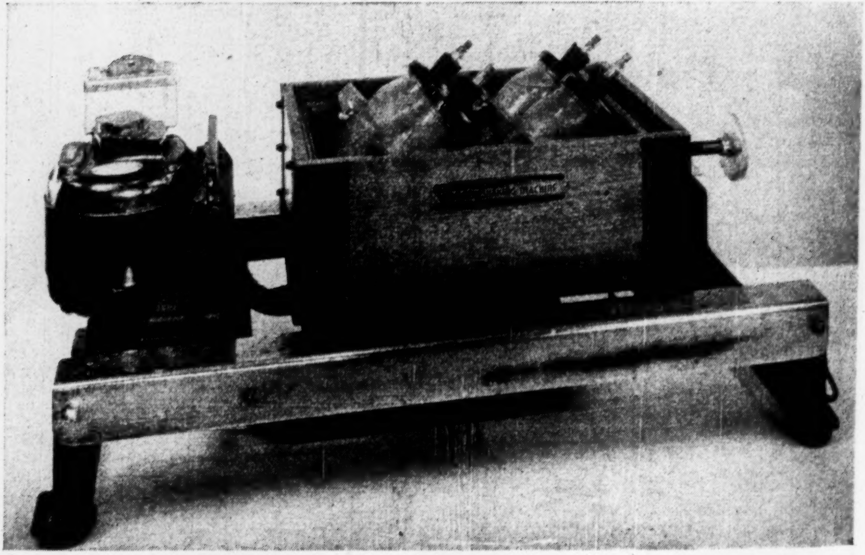

FIG. 8.—Arrangement for Shaking Individual Sulfonation Flasks.

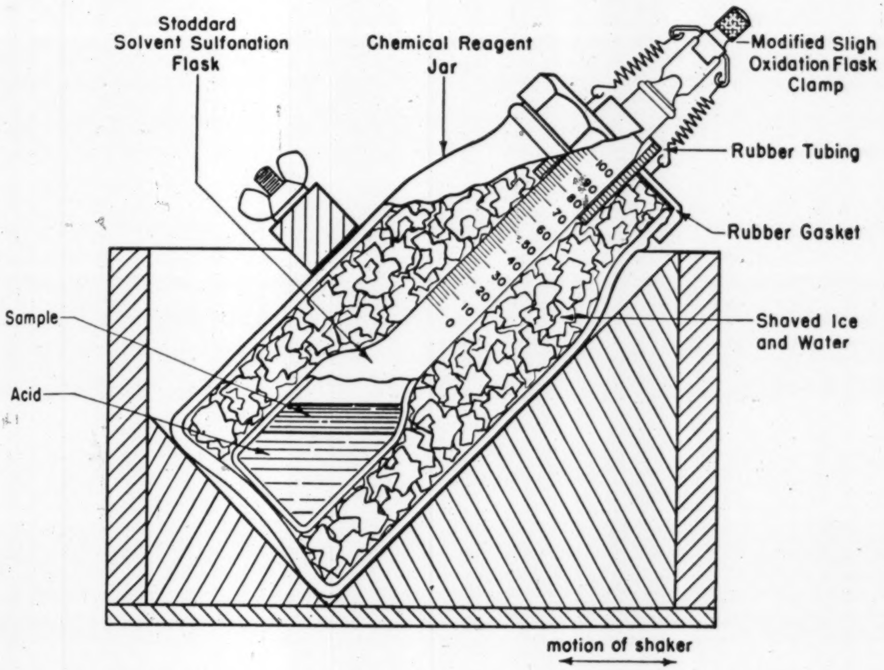
Stoddard Solvent Sulfonation Flask
Chemical Reagent Jar
Modified Sligh Oxidation Flask Clamp
Rubber Tubing
Rubber Gasket
Sample
Shaved Ice and Water
Acid
0
10
20
30
40
50
60
70
80
90
100
motion of shaker

FIG. 9.—Ice-Water Bath and Clamp Arrangement for Individual Sulfonation Flask.

REPORT OF COMMITTEE D-3*
ON
GASEOUS FUELS

A joint meeting of Committee D-3 on Gaseous Fuels and its seven subcommittees was held in Atlantic City, N. J., on October 6, 1946. A meeting of its Advisory Committee was also held on the same date.

RECOMMENDATION ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1946 Annual Meeting, Committee D-3 presented to the Society through the Administrative Committee on Standards the proposed Tentative Method of Test for Calorific Value of Gaseous Fuels by the Water Flow Calorimeter. This recommendation was accepted by the Standards Committee on September 9, 1946, and appears in the 1946 Book of A.S.T.M. Standards, Part III-A, under the designation D 900 - 46 T.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Collection of Gaseous Samples (S. J. Modzikowski, chairman).—A proposed Tentative Method for Sampling Liquefied Petroleum Gases has been completed and copies distributed for review and criticism. It is proposed to include comments received in a final draft for approval of the members of the subcommittee.

Preparation of a new method for sampling natural gases is now in progress. This will be completed in time for presentation to subcommittee members for their review prior to the Annual Meeting.

Subcommittee II on Measurement of Gaseous Samples (H. S. Bean, chairman).—A tentative draft of methods of measuring samples with brief references to methods of measuring larger volumes has been distributed to the subcommittee members for review and comment.

Subcommittee III on Determination of Calorific Value of Gaseous Fuels (R. S. Jessup, chairman).—Several changes in the specifications for calorimeter thermometers and wet meter thermometers proposed by Section A on Thermometers of Committee E-1 on Methods of Testing were approved at the meeting of the subcommittee on October 6, 1946, for later inclusion in Tentative Method D 900. Other suggested changes in its text will be submitted for consideration at the next meeting.

Samples of methane and propane for use with a recording calorimeter study have been obtained and analyzed by the mass spectrometer. Analyses showed the propane is practically pure but the methane contains so much impurities that its heating value is believed uncertain by about 0.5 per cent. Arrangements have been made for securing another supply.

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (E. F. Schmidt, chairman).—Arrangements have been completed with the National Bureau of Standards to formulate a tentative code and operating procedure for each specific gravity instrument, with certain tolerances for a limited range of specific gravity. This code will represent results of analysis of a very large amount of data obtained by the Bureau during the exhaustive tests of all specific gravity apparatus conducted some time ago.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

It is the hope of the subcommittee that formulation of this code may be completed during the year.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (A. W. Gauger, chairman).—Further studies have been conducted on the use of a water vapor detector, the principle of operation of which is based on change in conductivity of a phosphoric acid film as affected by the absorption of moisture. These will serve to supplement experiences previously obtained by the use of this instrument.

Subcommittee VII on Complete Analysis or Chemical Composition of Gaseous Fuels (Martin Shepherd, chairman) has issued three reports on cooperative analysis of standard samples. The first, already published, covers volumetric analysis by absorption and combustion and is entitled "Analysis of a Standard Sample of Natural Gas by Laboratories Cooperating with the American Society for Testing Materials." The second is entitled "Cooperative Analysis of a Standard Sample of Natural Gas with the Mass Spectrometer." This has been submitted to committee members and to the cooperating laboratories for study and is scheduled for publication.

The third report, intended for early publication, is entitled "Comparison of the Analysis of a Natural Gas by Volumetric Chemical Methods and by the Mass Spectrometer." As a result, a tentative method for the determination of the complete composition of natural gas by the mass spectrometer is now in preparation. A fourth report now being prepared will compare the analysis of a standard sample of natural gas by two volumetric-chemical methods. It will probably be used as a basis for a tentative method based on the chemical approach.

This report has been submitted to letter ballot of the committee which consists of 34 members; 26 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. W. GAUGER,
Chairman.

R. M. CONNER,
Secretary.

REPORT OF COMMITTEE D-4*

ON

ROAD AND PAVING MATERIALS

Committee D-4 on Road and Paving Materials has held two meetings during the past year; in Buffalo, N. Y., on June 27, 1946, and in Cincinnati, Ohio, on March 27, 1947.

During the year the committee lost, by the death of Charles N. Forrest on December 2, 1946, a member who has been one of the most valuable, respected and loved members of Committee D-4 substantially throughout its existence. With Committee D-8 on Bituminous Waterproofing and Roofing Materials, the committee has adopted a Joint Resolution in memory of Mr. Forrest, which reads as follows:

CHARLES NEEDHAM FORREST

In the death of Charles Needham Forrest on December 2, 1946, the Society has lost one of its most valuable, respected, and loved members. He was a member of the American Society for Testing Materials from 1901 until his death, a period of over 45 years of continuous membership. During this time, he was active in the work of Committees D-1, D-2, D-4, D-8, E-1, and E-8. He occupied several important offices in the Society; as a Member of the Executive Committee (now Board of Directors) from 1933 to 1935, as Chairman of Committee D-4 from 1942 to 1944, and as Vice-Chairman of Committee D-8 from 1918 to 1937. He was a member of the Philadelphia District Committee from 1935 to 1938.

Charles N. Forrest was born September 30, 1873 in Baltimore, Md. He graduated from the Maryland Institute and later attended Columbian University (now George Washington University), specializing in Chemistry. He started his professional career in the Test Department of the Baltimore and Ohio Railroad Company, becoming Principal Assistant Chemist; and in 1896 he entered the services of the Southern Railroad Company as Chemist at Washington, D. C. Later he became Chemist and Inspector for the Long Island Railraod Company until 1904, meanwhile being active on the Port Washington Extension Project as well as the Atlantic Avenue Improvement Project, Brooklyn, N. Y., finally serving as Engineer of Testing and Assistant Purchasing Agent of the Long Island Railroad. In 1904 he became associated with the New York Testing Laboratory as Chief Chemist, and in 1907 was in full charge of the design and control of street and highway paving and materials entering this field, particularly asphalt. When this laboratory was taken over by the Barber Asphalt Company, he continued with that corporation until his retirement under the pension plan in 1941, having served as Manager of the Technical Bureau taking over entire charge of supervision of paving operations and plant operation of both asphalt and oil refining. In 1927 he made a world tour in the interests of paving operations in various countries.

In addition to the American Society for Testing Materials, Charles N. Forrest was a member of the American Society of Civil Engineers, American Chemical Society, Society for Chemical Industry, Franklin Institute, the Association of Asphalt Paving Technologists, the Society of Municipal Engineers, and Fellow of the American Association for the Advancement of Science. He served as Chairman of the Advisory Research Committee, Asphalt Roofing Industry Bureau, continuing in this work several years after retirement from active corporate work.

His placid and pleasant personality, his wit and humor won for him many friends in and out of his profession at home and abroad. These characteristics, along with his ability to explain the technical necessities, many times saved the situation in a committee when an impasse developed.

Be It Therefore Resolved that this token of appreciation for his life and services be sent to Charles Forrest's wife and family and to the

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* **Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.**

President of the American Society for Testing Materials; and that it be recorded in the minutes of the Committees on Road and Paving Materials, and on Bituminous Waterproofing and Roofing Materials.

I. NEW TENTATIVE METHODS

Committee D-4 joins with Committee D-18 on Soils for Engineering Purposes in recommending that the Tentative Method of Test for Shear Strength of Flexible Road Surfaces, Subgrades and Fills by the Burggraf Shear Apparatus, as published by the Society in "Procedures for Testing Soils," September, 1944, page 119, be accepted for publication as tentative.[1] The application of this method is described in a paper by Mr. Fred Burggraf published by the American Road Builders' Association in 1940 as *Bulletin No. 70.* This paper presents data obtained in field tests and discusses the significance and interpretation of the data. The committee believes that promotion of this method of test should prove of direct value in the study of soil stabilization.

Committees D-4 and D-18 also jointly recommend that the Tentative Method of Test for Soil-Bituminous Mixtures, appended hereto,[1] be published as tentative. This method has been prepared by a group representing producers of typical bituminous materials used for the stabilization of highway soils. Its development and application were discussed in a paper presented at the 1946 Annual Meeting by E. O. Rhodes and P. F. Phelan.[2]

II. TENTATIVE REVISIONS OF STANDARDS

Committee D-4 recommends that the tentative revision of the Standard Method of Test for Penetration of Bituminous Materials (D 5 – 25), appended hereto,[1] be accepted for publication as tentative. Subcommittee B-24 on Penetration Test (Bailey Tremper, chairman) has made a detailed study of the standard method in connection with many objections to limitations in apparatus or procedure submitted with suggestions for improvement of the requirements. The recommended revision includes modified or more specific requirements designed to improve the reproducibility and reliability of the method.

Committee D-4 joins with Committee D-8 on Bituminous Waterproofing and Roofing Materials in recommending that a tentative revision of the Standard Methods of Testing Emulsified Asphalts (D 244 – 42)[3] be accepted for publication as tentative. The recommended revision provides an additional method for determination of residue as an alternate to the distillation method, and it is presented as tentative with a view to ultimate incorporation in the standard. The evaporation method has been compared with the distillation method in cooperative tests conducted by Subcommittee B-17 on Emulsion Tests (R. R. Thurston, chairman) and data supporting the recommendation are presented in Tables I, II, and III, showing percentages, penetration and ductility of residue obtained by the proposed evaporation and the present standard distillation methods. The proposed tentative revision is as follows:

TABLE I.—PERCENTAGE RESIDUE OBTAINED BY DISTILLATION *versus* EVAPORATION METHOD.

Emulsion	Distillation				Evaporation			
	New Jersey	Texas	Shell	Texas Co.	Maryland	Texas	Public Roads	Bitumuls
A, Quick setting...	57.0	57.5	57.2	58.0	58.0	57.7	57.7	57.4
B, Quick setting...	61.5	62.7	62.2	63.0	63.1	62.7	62.6	62.6
C, Quick setting...	60.5	...	60.6	60.5	60.8	...	61.3	60.4
D, Quick setting...	60.5	60.9	61.7	62.0	62.7	61.9	61.9	61.6
E, Mixing.........	62.0	61.3	62.0	62.5	63.1	62.0	62.6	62.6
F, Mixing.........	64.0	63.3	64.4	65.0	65.6	64.7	64.7	64.6
G, Mixing.........	56.0	...	56.8	57.0	58.5	...	57.3	56.8
H, Mixing.........	61.0	61.3	61.6	63.0	62.0	61.9	61.8	62.0

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[1] These methods were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part II.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1416 (1946).

[3] 1946 Book of A.S.T.M. Standards, Part II.

Residue by Evaporation.—Add the following method for determination of residue by evaporation as an alternate to the present distillation method:

RESIDUE BY EVAPORATION

41. *Apparatus.*—The apparatus shall consist of the following:

(*a*) *Glass Beakers.*—600-ml. low-form glass[a] beakers.

TABLE II.—PENETRATION OF RESIDUE OBTAINED BY DISTILLATION *versus* EVAPORATION METHOD.

Source	Emulsion No.	Bitumuls		Shell		Ohio		Tennessee	
		Distillation	Evaporation	Distillation	Evaporation	Distillation	Evaporation	Distillation	Evaporation
California, Quick setting	B-19-A	165	153	166	144	162	148	163	118
California, Mixing......	B-19-B	49	45	53	42	49	44	56	40
Venezuela, Quick setting	B-20-A	140	117	142	111	143	125	149	118
Venezuela, Mixing......	B-20-B	136	120	149	129	142	120	158	114
Talco, Quick setting.....	B-21-A	139	127	157	129	147	130	157	118
Talco, Mixing..	B-21-B	145	122	161	124	151	134	...	128

TABLE III.—DUCTILITY OF RESIDUE OBTAINED BY DISTILLATION *VERSUS* EVAPORATION METHOD.

All results are in centimeters.

Source	Emulsion No.	Bitumuls		Shell		Ohio		Tennessee	
		Distillation	Evaporation	Distillation	Evaporation	Distillation	Evaporation	Distillation	Evaporation
California, Quick setting..........	B-19-A	130	150+	111+	111+	100+	100+	85	94
California, Mixing................	B-19-B	150+	150+	111+	111+	100+	100+	105+	105+
Venezuela, Quick setting..........	B-20-A	140	139	111+	106	100+	100+	105+	105+
Venezuela, Mixing	B-20-B	101	89	100	68	100+	37	94	41
Talco, Quick setting..............	B-21-A	113	81	105	82	100+	91	98	105+
Talco, Mixing....................	B-21-B	110	83	106	100	100+	52	...	41

(*b*) *Glass Rods.*—Glass rods $\frac{1}{4}$ in. in diameter, 7 in. in length, with flame-polished ends.

(*c*) *Balance.*—A balance capable of weighing 500 g. to ± 0.1 g.

(*d*) *Oven.*—A thermostatically controlled oven, capable of maintaining a temperature of 325 ± 5 F. (163 ± 2.8 C.) as described in the Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6).

(*e*) *Sieve.*—A No. 50 (297-micron) sieve conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11).

42. *Procedure A* (*a*).—Procedure A shall be used when determination of residue only is required.

(*b*) Weigh exactly 50 g. of thoroughly mixed emulsified asphalt, representative of the sample, into each of three beakers, each beaker and a glass rod having previously been tared. Place the beakers containing the rod and sample in the oven, the temperature of which has been adjusted to 325 ± 5 F. (163 ± 2.8 C.) for 2 hr. Remove each beaker and stir the residue thoroughly. Replace in the oven for 1 hr., then remove the beakers from the oven, allow to cool to atmospheric temperature, and weigh.

(*c*) Calculate the percentage residue on each beaker as follows:

$$\text{Residue, per cent} = \frac{A - B}{C} \times 100$$

where:

A = weight of beaker, rod, and residue,

B = weight of tare beaker and rod, and

C = weight of sample.

(*d*) *Report.*—Report the residue by evaporation as the average of the three results.

43. *Procedure B* (*a*).—Procedure B shall be used when tests on residue from emulsion are required, in addition to percentage residue.

(*b*) Follow the procedure as described in Section 42 (*b*), except dehydrate four 50-g. samples. After the residue is determined and calculated, replace the beakers in the oven until the asphalt residue is sufficiently fluid to pass through a No. 50 sieve (usually requiring 15 to 30 min.). Then pour the residue through the No. 50 (297-micron) sieve and into suitable containers and molds for making such tests as desired, as described in Sections 36 to 40.

NOTE.—As the foregoing method tends to give an asphaltic residue lower in penetration and ductility than the distillation method as described in Section 5, material may be accepted but shall not be rejected as failing to meet specifications containing requirements for determination of residue by distillation on data obtained by evaporation. If residue from evaporation fails to meet requirements for properties specified for residue from distillation, tests shall be rerun using the distillation method. (Usually, however, results by evaporation do fall within requirement limits set for residue by distillation.)

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[a] Pyrex glass is very satisfactory for this purpose.

Committee D-4 joins with Committee C-9 on Concrete and Concrete Aggregates in recommending a tentative revision of the Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 - 46) as presented in the report of Committee C-9,[4] subject to favorable letter ballot of Committee D-4.

III. REVISION OF STANDARD, IMMEDIATE ADOPTION

Committee D-4 joins with Committee C-9 on Concrete and Concrete Aggregates in recommending for immediate adoption revision of the Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 - 46). The proposed revision is covered in the C-9 Report[4] and is substantially editorial in nature, providing for a general reference to the Standard Specifications for Sieves for Testing Purposes (E 11), drying of the test sample, and a preliminary separation of the sample after discharge from the machine by use of a sieve coarser than No. 12. The committee accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society.

IV. EDITORIAL CHANGE IN STANDARD

Committee D-4 joins with Committee D-8 on Bituminous Waterproofing and Roofing Materials in recommending that a section entitled "Outline of Methods" be inserted in Standard Methods of Testing Emulsified Asphalts (D 244 - 42) as an editorial revision.[5]

V. ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following three tentatives, with revisions in one specification as indicated, be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Specifications for Standard Sizes of Coarse Aggregate for Highway Construction (D 448 - 42 T), without revision,

Tentative Specifications for Tar (D 490 - 43 T), without revision, and

Tentative Specifications for Crushed Stone, Crushed Slag, and Gravel for Bituminous Concrete Base and Surface Courses of Pavements (D 692 - 42 T), revised as follows:

Section 5 (*a*).—Delete footnote *a*, applied to "Size No. 5" for use in open-mix bituminous concrete base course; this revision will permit the use of 75 per cent crushed gravel for Size No. 5 as well as other sizes specified for open-mix bituminous concrete base course. Add "Size No. 67" to the list of sizes permitted for use in closed-mix bituminous concrete base course.

Committee D-4 also joins with Committee D-18 in recommending that the Tentative Method of Test for Cement Content of Soil-Cement Mixtures (D 806 - 44 T) be approved for reference to letter ballot of the Society for adoption as standard, without revision.

VI. REVISION OF TENTATIVE

The committee recommends that the Tentative Method of Test for Hot Extraction of Asphaltic Materials and Recovery of Bitumen by the Modified Abson Procedure (D 762 - 44 T) be revised as follows:

Section 2 (*b*).—Change to read: "(*b*) *Balance.*—A balance capable of weighing 5000 g. to an accuracy of 0.1 g."

Section 2 (*f*).—Move Item (*11*) to precede present Item (*1*) and renumber as (*1*). Renumber Item (*1*) as (*2*). Renumber Item (*7*) as (*3*) and move to appropriate position. Combine Items (*2*), (*3*), and (*4*) as (*4*). Redesignate Item (*8*) as (*7*) and Item (*10*) as (*8*). Renumber Items (*12*), (*13*), (*14*), and (*15*) as (*9*), (*10*), (*11*), and (*12*), respectively.

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[4] See p. 246.

[5] This editorial change was made in the method as published in the special compilation, "A.S.T.M. Standards on Petroleum Products and Lubricants," October, 1947.

Change present Item (*13*) to read: "(*10*) A gas flow meter as shown in Fig. 2, or any type capable of indicating a gas flow up to 1000 ml. per min." Change present Item (*14*) to read: "(*11*) A gas inlet tube."

Fig. 2.—Distillation Assembly.—Change description applied to gas inlet tube to read: "3 mm. I.D. Copper Tubing Bent on $1\frac{3}{16}$-in. radius—having 3 holes $\frac{1}{32}$ in. drilled on $\frac{1}{2}$-in. centers along convex side of bend-end closed—first hole $\frac{1}{4}$ in. from end."

Section 3 (*a*).—Change to read: "(*a*) *Benzene* conforming to the Standard Specifications for Nitration Grade Benzene (A.S.T.M. Designation: D 835).

Section 5 (*a*).—Combine the last two sentences to read: "The stirrer shall be turned by hand one half turn every 15 min. (after the benzene becomes straw colored) to break up any settled filler and remove the last traces of bitumen."

Section 5 (*e*) *and* (*f*).—Combine to read: "(*e*) The flame shall be removed, the carbon dioxide gas shut off, and the apparatus disassembled. The contents of the flask shall then be poured into the 6-oz. container and cooled for further testing."

VII. TENTATIVES CONTINUED WITHOUT REVISION

The subcommittees of Committee D-4 and the joint subcommittee of Committees D-4 and D-18 have reviewed existing tentative specifications and methods of test which have been published by the Society for two years or more without revision, and the committee recommends that the following be continued as tentative, without revision:

Tentative Specifications for:

Materials for Stabilized Base Course (D 556 - 40 T), and Materials for Stabilized Surface Course (D 557 - 40 T). Various suggestions for changes in these two specifications have been made, which are under consideration by the joint subcommittee of Committees D-4 and D-18.

Calcium Chloride (D 98 - 46 T).

Tentative Methods of Test for:

Loss on Heating of Oil and Asphaltic Compounds (D 6 - 39 T). The method is in general use, but the committee believes that its provisions can be improved and should be studied by a recently organized subcommittee.

Moisture-Density Relations of Soils (D 698 - 42 T). Revisions of this method are under consideration by the joint subcommittee of Committees D-4 and D-18.

Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials (D 75 - 46 T),

Sampling Bituminous Materials (D 140 - 46 T),

Sampling and Testing Calcium Chloride (D 345 - 46 T), and

Sulfonation Index of Road Tars (D 872 - 46 T).

Tentative Revisions of Standards:

Specifications for Preformed Expansion Joint Fillers for Concrete; Nonextruding and Resilient Types (D 544 - 41), and

Methods of Testing Preformed Expansion Joint Fillers for Concrete, Non-extruding and Resilient Types (D 545 - 41). Subcommittee D-3 on Expansion Joint Materials has recently been very active and expects in the near future to recommend several revisions of the present standards and modifications in the existing tentative revisions of these standards.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

VIII. ACTIVITIES OF SUBCOMMITTEES

Of general interest are the activities of subcommittees with work now in progress which can be expected in the near future to result in recommendations for the approval of new tentatives and important revisions of existing standards.

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee B-3 on Distillation Tests (H. P. Ferguson, chairman) has concluded several series of cooperative tests to study modifications of the Standard Method of Test for Distillation of Cut-Back Asphaltic Products (D 402 - 36), with the objectives of providing for tests of all types of cutback in accordance with requirements of the revised method, minimizing foaming difficulties, and, at the same time, obtaining the same test results with the revised method as would be obtained by the present standard method.

Subcommittee B-6 on Extraction and Recovery of Constituents from Bituminous Mixtures (R. R. Thurston, chairman) is conducting cooperative work on modifications of the Tentative Method of Test for Hot Extraction of Asphaltic Materials and Recovery of Bitumen by the Modified Abson Procedure (D 762 - 44 T) involving the use of sieve cloth with larger openings.

Subcommittee C-1 on Asphalt Cements (F. V. Reagel, chairman) has prepared proposed specifications for asphalt cements which have been submitted to the committee for appropriate action for publication of the specifications as tentative. For many years the Society has not published specifications covering these road and paving materials.

This report has been submitted to letter ballot of the committee, which consists of 127 members; 78 members returned their ballots, of whom 59 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

W. J. Emmons,
Chairman.

B. A. Anderton,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:
  Hot-Mixed, Hot-Laid Asphaltic Concrete Base and Surface Courses (D 947 - 47 T), and
  Asphalt Cements for Use in Pavement Construction (D 946 - 47 T).
Tentative Revision of Standard Method of Test for:
  Distillation of Cut-Back Asphaltic Products (D 402 - 46).

These recommendations were accepted by the Standards Committee on September 4, 1947, and the new tentatives and tentative revision appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part II.

REPORT OF COMMITTEE D-5*

ON

COAL AND COKE

Committee D-5 on Coal and Coke held meetings in Buffalo, N. Y., on June 27, 1946, and in Philadelphia, Pa., on February 25, 1947. The Advisory Subcommittee held a meeting in Philadelphia, Pa., on February 25, 1947.

During the year nine members were added to the committee and three members resigned, resulting in the present membership of 59, of whom 15 are classified as producers, 21 as consumers, and 23 as general interest members.

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Method of Sampling Coal for Analysis (D 21 - 40) as this method is superseded by the Standard Methods of Sampling Coals Classed According to Ash Content (D 492-46).

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the annual meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Testing (W. A. Selvig, chairman) started an investigation of the suitability of the Brabender semi-automatic moisture tester for determining moisture in coal and coke. This work is being conducted in the Coal Analysis Laboratory of the U. S. Bureau of Mines. The investigation will be completed this coming year.

The subcommittee has under consideration the standardization of a weathering or slacking test for the lower rank coals and experimental work on this project is to be conducted in the laboratories of the Subbituminous Coal and Lignite Section of the Bureau of Mines at Golden, Col.

A section under the chairmanship of O. W. Rees has been formed to revise the present standard methods for determination of carbon, hydrogen, and nitrogen.

Subcommittee II on Nomenclature and Definitions (A. W. Gauger, chairman) has under consideration the preparation of definitions of terms pertaining to kinds of water in coal and has tentatively proposed to classify moisture into two types, namely, free moisture and bound or inherent moisture. Other definitions under consideration are terms pertaining to swelling or expansion of coal in connection with coal carbonization and coal combustion.

Subcommittee VII on Pulverizing Characteristics of Coal (John Van Brunt, chairman) is investigating methods for determining surface area of coal particles by means of measurement of resistance to air flow. Such methods, if satisfactory, would shorten the procedure given in the Tentative Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 37 T) which at present specifies sieving methods for measuring surface area of coal particles. The tentative methods for coal grindability, the Ball-Mill Method (D 408) and the Hardgrove Machine Method (D 409) are being continued as tentative pending this investigative work.

Subcommittee XIII on Coal Sampling (Henry F. Hebley, chairman) at a meeting held in Philadelphia, Pa., on February 24, 1947, recommended that the Standard Method of Sampling Coal for Analysis (D 21 - 40) be withdrawn. This method was first issued by the Society in 1916 but is now replaced by the Standard Methods of Sampling Coals Classed According to Ash Content (D 492 - 46) which was issued in 1938, revised in 1940 and 1945, and adopted as standard in 1946.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee XI on Coal Friability (R. E. Gilmore, chairman) was inactive during the past year but still has under consideration revisions of the Tentative Method of Drop Shatter Test for Coal (D 440 - 37 T).

Subcommittee XV on Plasticity and Swelling of Coal (C. C. Russell, chairman) plans on cooperating with the Committee on Stresses and Strains of the American Gas Association and the Committee on Coal Plasticity of the American Coke and Coal Chemicals Institute, as these committees have much of common interest. This cooperation is to be effected by overlapping memberships.

This subcommittee has under consideration a revision of the Standard Method for Free-Swelling Index of Coal (D 720 - 46) to provide for coke buttons which do not match the standard profiles. Procedures for providing for buttons not conforming to the standard profiles and which are used in the laboratories of the U. S. Bureau of Mines are given in the Bureau of Mines Report of Investigations 3989 entitled "An Investigation of a Laboratory Test for Determination of the Free-Swelling Index of Coal" by W. A. Selvig and W. H. Ode.

Subcommittee XVII on Significance of Tests of Coal and Coke (G. B. Gould, chairman) has been active during the year in furthering plans for preparing a publication on the significance of tests of coal and coke. It was agreed that experts should be invited to prepare statements on the significance of tests applicable to particular uses of coal and coke, such as (*1*) combustion, (*2*) carbonization, (*3*) complete gasification, (*4*) liquefaction, and (*5*) special uses. This material is to be used as the basis of preparation of an A.S.T.M. publication. Various sections of the subcommittee, each under a qualified chairman, have been formed to cover these uses of coal and coke.

The Section on Tests of Coal and Coke for Complete Gasification, E. S. Pettyjohn, chairman, held a meeting in Philadelphia, Pa., on February 24, 1947, at which time plans were made for the preparation of papers by qualified experts on the significance of tests for production of (*1*) producer gas, (*2*) water gas, and (*3*) synthesis gas.

Subcommittee XX on Sampling and Fineness Test of Pulverized Coal (J. B. Romer, chairman) held a meeting in Philadelphia, Pa., on October 23, 1946, and organized two sections. One section, under the chairmanship of J. Crites, is to work on sampling procedures, particularly for sampling coal from transport lines in which a mixture of air and pulverized coal is flowing. The other section, under the chairmanship of R. L. Blaine, will investigate sizing methods which will include dry and wet sieving methods and air-permeability methods for determination of specific surface of coal particles. When this work has been completed, it is proposed to prepare extensive revisions of the Standard Method of Sampling and Fineness Test of Powdered Coal (D 197 - 30).

This report has been submitted to letter ballot of the committee, which consists of 59 members, 49 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. FIELDNER,
Chairman.

W. A. SELVIG,
Secretary.

REPORT OF COMMITTEE D-6*
ON
PAPER AND PAPER PRODUCTS

Committee D-6 on Paper and Paper Products held one meeting during the year on June 27, 1946, at Buffalo, N. Y.

The Advisory Committee held one meeting during the year: on February 27, 1947. During this meeting a new Subcommittee III on Specifications for Paper was set up under the chairmanship of P. F. Wehmer.

Cooperative relations are being continued between Committee D-6 and other A.S.T.M. committees, as well as with other standardizing bodies.

At the present time, Committee D-6 consists of 81 members, of whom 65 are voting members; 25 are classified as producers, 27 as consumers, 23 as general interest, and 6 as consulting members.

I. New Tentative Methods

The committee recommends that the following five new methods, prepared by Subcommittee I, be accepted for publication as tentative, as appended hereto:[1]

Tentative Methods of Test for:
Bleeding Resistance of Asphalted Papers at Elevated Temperatures,
Copper Number of Paper and Paperboard,
Blocking Resistance of Paper and Paperboard,
Determining Titanium Dioxide in Paper, and
Crease Retention of Wrapping Paper.

II. Adoption of Tentative Methods as Standards

The committee recommends that the following four tentative methods be approved without change for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Test for:[2]
Adhesiveness of Gummed Tape (D 773 - 43 T),
Absorption by Bibulous Papers of Water and Writing Ink (D 824 - 45 T),
Degree of Wet Curl of Paper (D 826 - 45 T), and
Edge Tearing Strength of Paper (D 827 - 45 T).

III. Tentative Methods Continued as Tentative

The remaining tentative methods under the jurisdiction of Committee D-6, are recommended for continuation as tentative. Three of these are currently being reviewed.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

Activities of Subcommittees

Subcommittee I on Paper Testing Methods (C. G. Weber, chairman) has held two meetings during the year. The subcommittee has been active in developing new methods of test and has also continued its critical study of various methods of test of paper and paper products that have been adopted by other agencies, for example, those of the Technical Association of the Pulp and Paper Industry. The four tentative methods being recommended for adoption as standards are under the jurisdiction of this subcommittee.

Methods on the following subjects

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] These methods were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B.

[2] 1946 Book of A.S.T.M. Standards, Part III-B.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

are being given consideration by Subcommittee I:

Elongation,
Absorptiveness of paper to paraffin wax,
Linting,
Total chloride content of paper,
Analytical filter papers,
Brightness of paper,
Protinaceous nitrogen, qualitative and quantitative,
Acid-soluble iron in paper,
Reducible sulfur in paper,
Abrasion resistance,
Erasing quality,
Permanganate number,
Creasing for permeability tests,
Stiffness,
Gloss,
Fiber identification, and
Zinc pigments in paper.

In addition to the above subjects, studies are being made which may lead to proposed revisions in the Standard Methods of Test for Basis Weight of Paper and Paper Products (D 646 – 44), and Hydrogen Ion Concentration (pH) of Paper Extract (D 778 – 46), and in the Tentative Methods of Test for Water Vapor Permeability of Paper and Paperboard (D 783 – 44 T), and for Water Vapor Permeability of Paper and Other Sheet Materials at Elevated Temperature and Humidity (D 830 – 45 T).

Subcommittee I also has developed a method of test for siliceous grit content of paper and paper products. This is still under consideration and is, therefore, not being submitted for publication as tentative, but is included in Appendix I to this report as information to elicit criticism.

Subcommittee II on Significance of Test Methods (C. C. Heritage, chairman) held no meeting during the year. This subcommittee does not contemplate further meetings until such time as a revision in the monograph on "Paper and Paperboard, Characteristics, Nomenclature, and Significance of Tests," is necessary.

Subcommittee IV on Fiberboard and Fiberboard Containers (W. B. Lincoln, Jr., chairman) has held one meeting during the year. Test procedures for the following are under consideration:

Ring crush test,
Adhesion test of component parts of corrugated board,
Immersion test and delamination of plies,
Beam test,
Flat crush test, and
Bursting strength of paperboard.

In addition to the above subjects, studies are being made which may lead to proposed revisions in the Standard Methods of Test for Conditioning Paperboard, Fiberboard, and Paperboard Containers for Testing (D 641 – 43), and method A of the test for Thickness of Paper and Paperboard (D 645 – 43), and in the Tentative Method of Test for Puncture and Stiffness of Paperboard, Corrugated and Solid Fiberboard (D 781 – 44 T).

Subcommittee IV also has developed a method of test for scuff resistance of paperboard. This is still under consideration and is, therefore, not being submitted for publication as tentative, but is included in Appendix II to this report as information to elicit comment and criticism.

This report has been submitted to letter ballot of the committee, which consists of 65 voting members; 44 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. S. REID
Chairman.

G. H. HARNDEN,
Secretary.

APPENDIX I

PROPOSED METHOD OF TEST FOR THE SILICEOUS GRIT CONTENT OF PAPER AND PAPER PRODUCTS[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test covers the procedure for determining the siliceous grit content of paper and paper products that are used for purposes requiring punching and shearing operations. It is intended to be a measure of the abrasive properties, which may cause undesirably rapid dulling of punches and dies or of shear knives.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Beaker.*—A 1-liter, chemically resistant glass[2] beaker.

(*b*) *Filter Paper.*—Rapid, ashless filter paper.[3]

(*c*) *Filtering Funnel.*—A fluted, chemically resistant glass[2] Bunsen filtering funnel 65 mm. in diameter and having a 60-deg. angle.

(*d*) *Filter Cone.*—A platinum or paper filter cone.

(*e*) *Centrifuge Tube.*—A glass Goetz phosphorus tube,[4] pear-shaped with a ground-glass stopper, and having the stem graduated at the 25-, 50-, and 100-ml. marks and graduated from 0.0 to 0.2 ml. in 0.01-ml. divisions.

(*f*) *Porcelain Crucible.*—A high-form porcelain crucible, having a capacity of 10 ml., 30 mm. in diameter at the top and 25 mm. in height.

(*g*) *Bunsen Burner.*

(*h*) *Desiccator.*—A desiccator containing anhydrous calcium chloride.

Reagents.

3. (*a*) *Nitric Acid* (*fuming*).

(*b*) *Sulfuric Acid* (sp. gr. 1.84).

(*c*) *Distilled Water.*

Conditioning

4. The test specimen shall be conditioned in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685).[5]

Procedure

5. (*a*) Weigh out (Note 1) about 25 g. of the air-dry paper to the nearest 0.01 g. and place in a 1-liter beaker.

NOTE 1.—If the paper contains a soluble impregnant such as oil or paraffin, it should be extracted before weighing with a suitable solvent in accordance with the Standard Method of Test for Paraffin Content of Waxed Paper (A.S.T.M. Designation: D 590).[5]

(*b*) Add 100 ml. of fuming HNO_3 and warm on the hot plate until the paper becomes a thin paste; then boil gently until a low-viscosity solution results. Evaporate the solution to a volume of 30 to 40 ml. Cool, add 20 ml. of H_2SO_4 (sp. gr. 1.84), and heat until white fumes of SO_3 are evolved (Note 2). Clarify the solution by cautiously adding small volumes of fuming HNO_3, allowing the drops to run down the inside wall of the beaker, and reheat until SO_3 fumes are evolved. Repeat this clearing operation as often as is necessary to produce a clear solution that is a light straw color. When clear, give the solution a final fuming to remove the HNO_3.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products. Published as information, January, 1947, and June 1947.

[2] Pyrex glass is satisfactory for this purpose.

[3] Whatman No. 41 filter paper, or equivalent, is satisfactory for this purpose.

[4] Eimer and Amend Catalog No. 5-624, or equivalent, is satisfactory for this purpose.

[5] 1946 Book of A.S.T.M. Standards, Part III-B.

NOTE 2: *Caution.*—The digestion should be conducted under a hood. Because of the corrosive characteristics of the acids specified, they should be handled with the utmost care.

(*c*) Cool the solution to room temperature and dilute to 400 ml. with distilled water. Boil gently for several minutes, filter while hot through a rapid, ashless paper, and wash the residue with distilled water.

(*d*) Transfer the residue from the filter paper to a 100-ml. Goetz centrifuge tube, using distilled water for this purpose. Dilute the suspension to the 100-ml. mark with distilled water and, with the stopper in place, shake it vigorously by hand for about 1 min. Allow the suspension to settle partially under the force of gravity for exactly 10 min. at room temperature (22 to 28 C.). At the end of this settling period, siphon off and discard the supernatant suspension, being careful not to disturb the large particles that have settled in the stem of the tube. Again dilute to 100 ml. and repeat the shaking, settling, and siphoning operations three more times.

(*e*) After the fourth siphoning, record the volume of the particles collected in the stem of the tube and then add about 50 ml. of distilled water. Shake the mixture thoroughly, and quantitatively collect the particles on a rapid, ashless paper.

(*f*) Place the filter paper containing the particles in an ignited and weighed porcelain crucible. Dry the paper, and ignite over a Bunsen burner until all the carbon has been burned off. Allow the crubible to cool in a desiccator and weigh.

(*g*) Calculate the percentage of grit, based on the air-dry weight of the sample.

Report

6. The report shall include the following:

(*1*) The volume of grit determined in accordance with Section 5 (*e*), and

(*2*) The percentage of grit, calculated on the basis of the weight of the paper. For purposes of acceptance and rejection, the results shall be reported to the nearest 0.01 per cent; for other purposes, such as tests applicable to research, the results shall be reported to the nearest 0.001 per cent.

Precision

7. The precision of the method (numerical value of the standard deviation of individual determinations of the percentage of grit content made on a series of 25-g. test specimens having highly uniform grit content) is approximately 0.002.

APPENDIX II

PROPOSED METHOD OF TEST FOR SCUFF RESISTANCE OF PAPERBOARD[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method covers the test for scuff resistance of paperboard. The test is intended to measure scuffing, which is the actual lifting of the surface of the paper formation and its subsequent rolling into a "quill," rather than the production of ground or powdered fibers. The test is not intended to measure abrasion.

Apparatus

2. The apparatus shall consist essentially of a power driven testing machine having a reciprocating arm to which is attached a smooth flat plate free to slide over a fixed plate. Both plates shall be equipped with clamps capable of holding taut the test specimen in such a manner that the corresponding surfaces of two specimens are held flat against each other. The upper or reciprocating plate shall have a total weight of 12 lb. (Note). The leading edges of both plates shall be beveled or rounded away from the specimens to avoid the presence of a square edge moving across the surface of the specimens. It is recommended that both plates be covered with a thin layer of sponge rubber to provide a backing for both upper and lower specimens. It is found that this insures a more uniform contact of the two specimens than a backing of nonresilient material. The drive mechanism shall be such as to cause the arm and moving plate to reciprocate at a constant rate of 60 double strokes per minute, a double stroke being the forward plus the backward movement of the plate, or one full cycle of this mechanism. The total distance of travel of the moving plate shall be 6 in. in each direction. In order to minimize the tendency to rotate about the leading edge of the moving plate, the connection between the driving arm and the moving plate should be as close as possible to the plane of the specimen. The apparatus shall be provided with a counter to indicate at all times the number of double strokes produced.

NOTE.—If it is necessary to test specimens smaller than those prescribed in Section 3, the weight of the upper plate shall be adjusted so as to provide a pressure equal to 0.5 psi. of constant contact area. Constant contact area is the area of the lower (fixed) specimen that is at all times in contact with the moving specimen; for example, the full-size 12 by 6-in. specimen, which provides a 10 by 6-in. exposed surface for scuffing, has a constant contact area of 24 sq. in.

Test Specimens

3. Not less than 20 representative specimens measuring 12 by 6 in. shall be cut from the sample. One half of the specimens shall be cut with the 12-in.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products. Published as information, June, 1947.

sides parallel to the grain direction and the other half shall be cut with the 6-in. sides parallel to the grain direction.

Procedure

4. (*a*) At least ten pairs of specimens shall be tested, one half with the grain direction parallel to and the other half with the grain direction at right angles to the direction of the stroke. In all cases, the grain direction shall be parallel in the two specimens in each pair.

(*b*) Two specimens shall be tested simultaneously, one being rubbed against the other with corresponding surfaces in contact. The specimens shall be clamped to the plates of the testing machine in such a manner as to leave clear a flat exposed surface 10 by 6 in., with the 10-in. dimension parallel to the direction of the stroke.

(*c*) With the two specimens clamped in place and their surfaces in contact, the testing machine shall be started and operated until failure of one or both of the two specimens results (Note 1). Failure shall be considered to have occurred when surface fibers in formation are lifted from the main body of the sheet (Note 2).

NOTE 1.—This method is intended primarily for testing liner board that is to be converted into corrugated or solid fiber containers. Occasionally such liner board shows a distinct tendency to peel or form quills on scuffing, and this tendency has been noticed on the corrugated combiner, on roller conveyor systems in the shippers plant, and in freight cars where one box rubs against another. Most liners, however, do not exhibit this tendency, so that it is not the intention in this method to carry the test to the quill forming stage or to final failure regardless of how long this takes. Usually a relatively short time under test is sufficient to disclose a tendency to peel. If the specimen does not show this tendency after 100 double strokes, the test should be discontinued and reports should show that this specimen is not susceptible to scuffing. If the specimen does show a tendency to peel in the early stages of the test, the test shall be carried on to failure of the specimen.

NOTE 2.—The end point is not always clear-cut, and the type of failure will vary with different types of materials. Usually it takes the form of a quill of fibers peeled from the surface. The appearance of loose fibers in the form of dust shall not be considered failure. The fibers must be in formation to constitute failure. The test should be stopped at intervals to permit inspection of the specimens. The frequency of these inspections will necessarily vary, depending upon the expected scuff resistance of the material under test. Loose fiber particles should be removed from the specimens by lightly brushing the surfaces at intervals of ten double strokes to prevent rolling or balling.

Report

5. The report shall include the following:

(*1*) Number of double strokes required to produce failure,

(*2*) Exposed area of the specimens, and

(*3*) Pressure in pounds per square inch of constant contact area (Note, Section 2).

REPORT OF COMMITTEE D-7[*] ON WOOD

Committee D-7 on Wood held meetings at Buffalo, N. Y., on June 27, 1946, in conjunction with the Annual meeting of the Society, and at Chicago, Ill., on March 18, 1947, at the time of the American Railway Engineering Association Convention.

Committee D-7 has shown a substantial increase in membership during the past two years, in conjunction with the broadening of the scope, the organization of a number of new subcommittees, and reactivation of certain phases of the work. This organization program will be further developed during the coming year. Included in the plans are the recruitment of new members to broaden consumer representation, the completion of the membership of Subcommittee VII on Wood Poles and Cross Arms, and the appointment of subcommittee chairmen to fill present vacancies.

I. Adoption of Tentative as Standard

The committee recommends that the Tentative Methods of Testing Plywood, Veneer, and Other Wood and Wood-Base Materials (D 805 - 45 T)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

II. Revision of Standard and Reversion to Tentative

The committee recommends that the Standard Methods of Testing Small Clear Specimens of Timber (D 143 - 27) be revised and reverted to tentative, as appended hereto.[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

Activities of Subcommittees

Subcommittee I on Specifications for Timber (L. J. Markwardt, chairman).—Some phases of the work of this subcommittee, particularly the revision of the Standard Specifications for Wood Joist and Plank, Beams and Stringers, and Posts and Timbers (D 245 - 37) are awaiting a report on the analysis of present working stresses for timber, and the revision of Forest Products Laboratory *Miscellaneous Publication 185*, entitled, "Guide to the Grading of Structural Timbers and the Determination of Working Stresses."

The Standard Specifications for Round Timber Piles (D 25 - 37) will be reviewed, and revisions made if deemed desirable on the basis of suggestions received.

Subcommittee II on Laminated Timber (Frank J. Hanrahan, chairman).—During the past year the work of this subcommittee has been held up pending completion of extensive studies by the U. S. Forest Products Laboratory which are needed as a basis for proposed standards. These studies appear to be reaching a conclusion and it is anticipated that the subcommittee will have an active program during the coming year. It is contemplated that the first project undertaken will be the formation of standard procedures for testing glued laminated lumber, particularly the completed product, for use in determining whether the product meets such requirements as may be set up for various types of intended service, for example interior, exterior, or submerged use. The purpose would be to provide standard test procedures needed for control in manufacture and inspection of glued laminated timber.

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] 1946 Book of A.S.T.M. Standards, Part II.

[2] These revised methods were accepted by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part II.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee III on Plywood (J. A. Liska, chairman).—In considering the work of the committee in the plywood field, consideration was given to the formulation of general specifications for plywood. Such specifications would permit the purchaser or user to select material that would conform to his requirements and would promote economy through the use of lower-grade, lower-cost material when it was satisfactory for the intended use. As an approach to this problem, the subcommittee was circularized to determine its opinion as to the need and possible scope of such specifications. Two members questioned the need or possible usefulness of A.S.T.M. specifications for plywood, while four members believed that specifications setting up requirements for plywood for broad fields of specific use plus requirements for strength grades would be of definite value and that plans for the formulation of such specifications should be carried forward. In the next year it is hoped that agreement may be reached on this problem, and that work may be started on such specifications.

Subcommittee IV on Wood Paving Blocks (W. H. O'Brien, chairman) has developed a preliminary draft of specifications for creosoted end-grain wood block flooring for interior use. This draft is undergoing further revision to meet requirements found to be most satisfactory on the part of purchasers and manufacturers. It is planned to submit the specifications for publication as tentative during the coming year.

Subcommittee V on Methods of Preservative Treatment of Timber (A. L. Kuehn, chairman) is considering the advisability of revising and bringing up to date specifications on methods of preservative treatment so that they will conform with latest practices.

Subcommittee VI on Timber Preservatives.—The chairmanship of this subcommittee remains to be filled as a result of the recent resignation of W. P. Arnold. The work facing this subcommittee involves the preparation of specifications for extensively used preservatives other than those now covered by A.S.T.M. Standards, and a review of present standards and specifications as enumerated in the 1946 Report.[4]

Subcommittee VII on Wood Poles and Cross Arms.—This new subcommittee is being organized, and will be activated during the coming year. Aside from the question of pole specifications, three problems confronting the subcommittee are (*1*) development of standard methods of test for wood poles, (*2*) development of standard methods of tests for cross arms of different types, simulating the method of support and loading involved, and (*3*) preparation of specifications for cross arms, with special consideration to structural grades for cross arms containing knots or other features affecting strength.

Subcommittee VIII on Modified Wood and Wood-base Materials (J. A. Liska, chairman) is planning to survey the possible fields of service for the subcommittee during the coming year, as a background for planning an active program of work. Consideration is being given to the preparation of specifications for "compreg," the term used to designate resin impregnated compressed wood, which has developed a number of special uses.

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[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 385 (1946).

Subcommittee IX on Methods of Testing (L. J. Markwardt, chairman).—Principal progress during the past year was the revision of Standard Method D 143, which is appended hereto.[2] The revision provides for increasing the number of tests on air-dry material, improvement in the tension parallel to grain test specimen, some modification in methods of selecting and matching material, and certain improvements in technique, all developed to improve the standard and the effectiveness of the results without vitiating comparability of results with those obtained under the present Standard D 143 – 27. Active work is in progress on the development of a secondary standard for methods of testing wood in the form of specimens 1 by 1 in. in cross-section, as against the 2 by 2-in. size called for in Method D 143. The need for the secondary standard has developed because of the increasing importance of evaluating the properties of material from smaller trees, including second-growth, which do not provide adequate test specimens under the present method.

Subcommittee X on Nomenclature and Definitions.—During the coming year the chairmanship will be filled, and the revision of tree names in the Standard Definitions of Terms Relating to Timber (D 9 – 30) will be continued, and completed if possible.

Subcommittee XI on Moisture Content of Timber (M. E. Dunlap, chairman).—An article has been prepared for publication describing the development and use of the apparatus for determining the moisture content of wood treated with chemicals, such as are used in fire-retardant treatments. Improvement has been made during the year on the main problem in the use of the apparatus, namely that of accurately determining the dew point. It is felt that this feature has been solved satisfactorily, with the maximum deviation now being reduced to 1.3 per cent. Publication of this paper in the ASTM Bulletin is planned for the near future. Further work of the subcommittee will concern the problem of specifications for moisture meters.

Subcommittee XII on Fire-retardant Wood (W. H. Fulweiler, chairman).—A well-attended meeting of this subcommittee, with many visitors from the main committee, was held in Chicago, Ill., on March 18. It was decided to ask Committee E-5 on Fire Tests of Materials and Construction to give consideration to the preparation of a test suitable for plant use in measuring fire spread, and to ask all other individuals or organizations who have used the fire-tube test to make available experience data, especially in regard to char and flame spread.

This report has been submitted to letter ballot of the committee, which consists of 53 members; 38 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

HERMANN VON SCHRENK,
Chairman.

L. J. MARKWARDT,
Secretary.

REPORT OF COMMITTEE D-8[*]
ON
BITUMINOUS WATERPROOFING AND ROOFING MATERIALS

Committee D-8 on Bituminous Waterproofing and Roofing Materials held one meeting during the year, in Philadelphia, Pa., on February 27, 1947.

In December, 1946, the committee suffered the loss, through death, of one of its oldest and most valued members, Charles N. Forrest. Mr. Forrest had been a member of the Society since 1901 and was especially active in the affairs of Committee D-8 and Committee D-4 on Road and Paving Materials. A memorial resolution has been prepared by these two committees and a copy sent to his family.

All of the standards and tentatives for which Committee D-8 is responsible have been reviewed leading to the recommendations set forth in this report.

I. Revisions of Tentatives

Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets and Shingles (**D 228 - 44 T**).[1]—To clarify the application of these methods and improve their accuracy the following revisions are recommended:

Section 19 (*a*).—Change the first three sentences to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> From the sample representing the average weight of the lot of roofing as obtained in Section 9 or 15 [specimens shall be cut, each measuring approximately 2 in. in width by 8 in. in length], *die-cut specimens having a total area of at least 16 sq. in., measured to an accuracy of 0.5 per cent shall be prepared.* These specimens shall be weighed [the length and width measured to within $\frac{1}{32}$ in.], and the weight calculated per 108 sq. ft. Any of the [2 by 8-in.] specimens whose weight varies more than 1.5 per cent from the average weight of the lot as determined in Section 8 or 14 shall be rejected.

Section 19 (*b*).—Change the first sentence to read as follows by the omission of the words and figures in brackets: "Two of the weighed [2 by 8-in.] specimens shall each be separated into three horizontal sections approximately at the points indicated by the arrows *a* and *b* in Fig. 1."

Section 21 (*a*).—Change the second and third sentences to read as follows by the addition of the italicized words: "The insoluble material shall be removed *by filtering* and dried in air and the pieces of felt picked out and brushed free of adhering mineral matter. This felt shall then be placed in a tared weighing bottle, further dried at 105 to 110 C. (221 to 230 F.) for 30 min., *cooled in a dessicator*, and weighed."

Section 23.—Add the following sentence to the end of this section: "The total percentage of excess ash shall be calculated from the above data and used in determining the weight of desaturated dry felt per 108 sq. ft. as outlined in Section 24."

Section 24.—Change from its present form: namely,

> 24. The combined weights of the felt from the center portion obtained in Section 20 and the weights of the felts from the weather side and

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] 1946 Book of A.S.T.M. Standards, Part II.

reverse side portions obtained in Sections 21 and 22, respectively, corrected for excess ash (Section 23) shall be the total weight of dry felt in the two 2 by 8-in. specimens. From this weight calculate the weight of dry felt per 108 sq. ft.

to read as follows:

24. Two of the die-cut specimens of the finished product selected in accordance with Section 19 shall be extracted with carbon disulfide in a suitable extractor or centrifuge until the washings are colorless. The desaturated felt shall be dried in air, brushed free of any adhering mineral matter, then placed in a tared weighing bottle and further dried at 105 to 110 C. (221 to 230 F.) for 30 min., cooled in a desiccator, and then weighed. From this weight, calculate the weight of dry felt, corrected for excess ash, per 108 sq. ft. of finished roofing as outlined in Section 23.

Section 26.—Change the note at the end of this section from its present form: namely,

NOTE.—The method of analysis described in this section is not suitable for analyzing thick butt shingles of the "Tapered Type."

to read as follows:

NOTE.—In analyzing shingles or roofing of graduated thicknesses, samples shall be taken so as to give an average analysis of the area being tested.

Tentative Specifications for Asphalt for Dampproofing and Waterproofing (**D 449 - 42 T**).[1]—To eliminate asphalts which may slide excessively when applied to vertical surfaces in direct sunlight or exposed to temperatures above 125 F., and to bring the ductility in harmony with that expected on this type of material, the following changes are recommended:

Table 1.—For type C, change the minimum requirement for softening point (ring-and-ball method) from "170 F. to read "180 F. (82 C.)," and the minimum requirement for ductility at 25 C. (77 F.), (5 cm. per min.) from "2.5" to read "2."

II. ADOPTION OF TENTATIVES AS STANDARDS

Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (**D 146 - 44 T**).[1]—Since these methods cover sampling as well as testing, it is recommended that the following editorial changes be made and the methods be approved for reference to letter ballot of the Society for adoption as standard:

Title.—Add "Sampling and" and delete "of" from the first part of the title.

Section 1.—Add "sampling and" before "examination" in the first line of this section.

Tentative Specifications for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-Up Roofs (**D 250 - 44 T**) *and Asphalt-Saturated and Coated Asbestos Felts for Use in Constructing Built-Up Roofs* (**D 655 - 44 T**).[1]—These tentative specifications have been ready for adoption as standard for the past four years but this action was deferred until the Federal Specifications Board completed its studies of similar specifications. The proposed Federal specifications are now in agreement with these A.S.T.M. specifications and the committee therefore recommends that both of them be approved for reference to letter ballot of the Society for adoption as standard.

III. REVISION OF STANDARDS, IMMEDIATE ADOPTION

Standard Specifications for Asphalt-Saturated Roofing Felt for use in Waterproofing and in Constructing Built-Up Roofs (**D 226 - 44**),[1] and *Coal-Tar Saturated Roofing Felt for use in Waterproofing and in Constructing Built-Up Roofs* (**D 227 - 44**).[1]—In order to have these specifications conform to the new standards which have been agreed upon between the roofing industry and the

Division of Simplified Practice of the U. S. Department of Commerce and those which have been accepted by Underwriters' Laboratories and the Federal Specifications Board, it is recommended that in Section 9 of Specifications D 226 the minimum weights for the 15-lb. type and the 30-lb type be changed from "14" and "28 lb." to read "13" and "26 lb.", respectively; and in Section 8 of Specifications D 227, that the minimum weight be changed from "14" to read "13 lb." The committee requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

IV. TENTATIVE REVISION OF STANDARD

Standard Methods of Testing Emulsified Asphalts (**D 244 - 42**).[1]—This method is under the joint jurisdiction of Committees D-4 and D-8. Subcommittee B-17 of Committee D-4 has recommended as an alternate the addition of a test for "residue by evaporation."[2] Committee D-8 agrees that this addition is desirable and recommends, jointly with Committee D-4, that it be added as a tentative revision of Standard Methods D 244-42.

V. TENTATIVES CONTINUED WITHOUT REVISION

Committee D-8 recommends that the following tentatives be continued without change:

Tentative Specifications for:

Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 - 46 T) and Asphalt Roofing Surfaced with Mineral Granules (D 249 - 46 T).

Coal-Tar Pitch for Steep Built-Up Roofs (D 654 - 42 T). A study of slide test methods and requirements is now being made for possible inclusion in these specifications.

Tentative Recommended Practice for:

Accelerated Weathering Test of Bituminous Materials (D 529 - 39 T). A survey conducted in 1946 by Subcommittee VIII showed a wide variation in lamps and operating procedure. It is felt that better uniformity should be obtained before this practice is recommended for adoption as standard.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee which consists of 69 members; 52 members returned their ballots, of whom 49 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. S. MILLER,
Chairman.

H. C. HOWELL,
Secretary.

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[2] See p. 344.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

REPORT OF COMMITTEE D-9*

ON

ELECTRICAL INSULATING MATERIALS

Committee D-9 on Electrical Insulating Materials held three meetings during the year: in Atlantic City, N. J., on October 16 and 17, 1946, in Philadelphia, Pa., on February 27 and 28, 1947, and in Atlantic City on June 16 to 18, 1947.

At these meetings, actions were taken in which resulted in several new standards and revisions of existing tentatives. Some projects, which are still under way, are mentioned later in this report.

At the Atlantic City meeting in October, 1946, Subcommittee IV sponsored a Symposium on Insulating Oils comprising four papers. Three of the papers were published in the May, 1947, issue of the ASTM Bulletin[1] and the fourth will appear in a subsequent issue.[2]

This subcommittee also sponsored a second symposium held in connection with the 1947 Annual Meeting. The titles of the papers of both symposiums are listed later in this report under the activities of Subcommittee IV.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1946 Annual Meeting, Committee D-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:

Testing Electrical Insulating Oils (D 117 - 46 T), and

Testing Askarels (D 901 - 46 T).

Revision of Tentative Methods of:

Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 46 T), and

Testing Vulcanized Fibre Used for Electrical Insulation (D 619 - 46 T).

These recommendations were accepted by the Standards Committee on November 23, 1946, and the new and revised tentatives appear in the 1946 Book of A.S.T.M. Standards, Part III-B.

On December 31, 1946, the Standards Committee accepted the recommendation of Committee D-9 that the Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes be published as tentative. This new tentative appears in the 1946 Book of A.S.T.M. Standards, Part III-B bearing the designation D 902 - 46 T.

RECOMMENDATIONS AFFECTING STANDARDS

As a result of the year's work, the committee is submitting three new tentatives, revisions in six tentatives, tentative revision of one standard, and an editorial revision of a standard. These recommendations have been submitted to letter ballot[3] of the committee the results of which will be reported at the Annual Meeting.

ACTIVITIES OF SUBCOMMITTEES

The activities of the various subcommittees are briefly reviewed in the following summary of committee activities.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] ASTM BULLETIN, No. 146, May, 1947.

[2] ASTM BULLETIN, No. 149, December, 1947.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee I on Insulating Varnishes, Paints and Lacquers (P. E. Demmler, chairman) has reorganized its activities, providing four general sections on oxidizing, heat reactive, laminating, and high-temperature varnishes, respectively. Six other sections are provided to study test methods which are to be used in preparing standard methods.

The former sections on deep drying tests, set time tests, and heat endurance tests, are included in the above, and work on these subjects is continuing.

Subcommittee III on Plates, Tubes, Rods and Molded Materials (G. H. Mains, chairman).—During the past year new Tentative Specifications for Nonrigid Polyvinyl Tubing[4] have been prepared and referred to letter ballot of Committee D-9. Revisions in the Rockwell hardness methods in D 785 - 44 T and in D 229 - 46 have been accepted to make the method more universally applicable to various plastics. A revision of the impact strength method D 256 - 43 T has also been approved by the subcommittee. Revisions of D 229 and D 48 on flexural strength of sheets and molded materials have been incorporated; also minor revisions of the acetone extraction method D 494 - 41, and methods of testing vulcanized fiber D 619 - 42 T.

The subcommittee is actively working in cooperation with Subcommittee VI of Committee D 20 on Plastics in the preparation of specifications for electrical grades of thermoplastic materials, including polystyrene, polyvinyl chloride, polyvinylchloride acetate copolymer, and polytetrachlor-ethylene. The work on bonding strength has been transferred to Subcommittee I of Committee D-20, with members of Committee D-9 still taking an active part. This has resulted in a promising new method which is being submitted for subcommittee approval. The subcommittee is cooperating with Subcommittee XII in an investigation of arc resistance and insulation resistance methods. Some interesting results of tests in power factor at low and high temperatures have been reported, and the problem is being studied further. Some progress has been made on the development of a better method for tension test of tubes and rods in cooperation with Subcommittee I of Committee D-20.

Subcommittee IV on Liquid Insulation (E. A. Snyder, chairman) is continuing its close cooperation with Committee D-2 on Petroleum Products and Lubricants in work that is under way to improve the tests for neutralization and saponification numbers.

Correlation tests to determine the relation of the sludge test values as determined by Tentative Method D 670 - 42 T with actual service life of insulating oil in transformers have been continued during the year.

There are 24 laboratories represented in the activities of Section B. Of these, 20 laboratories are participating in the field tests on transformers to determine the behavior of transformer oil in service use. The fundamental tests involved which are required from all laboratories are the two A.S.T.M. sludge tests (sludge accumulation and high-pressure bomb), color, and acidity. In addition, several laboratories are including other data on the various oils such as interfacial tension test, power factor, steam emulsion, resistivity, water, etc.

The transformers involved in the field tests of Section B are located at plants of the New England Power, American Gas, Detroit Edison, and Commonwealth Edison Co. The transformers at New England Power include reconditioned oil. The transformers at Detroit Edison include three grades of oil described as (*1*) duplicating normally good transformer oil, (*2*) a medium grade transformer oil gaged on the basis of laboratory sludge tests, and (*3*) a highly sludging transformer oil gaged on the basis of laboratory sludge tests. Of these the highly sludging oil appears to be deteriorating, thereby substantiating the preliminary laboratory sludge test classification.

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[4] These methods and specifications were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B.

In general, the tests of transformers with new oil have not proceeded sufficiently far to warrant any final decision. In some instances the transformers on test indicate a falling value of sludge formation on the laboratory test. This is not explainable. No attempt at explanation will be made until more data have accumulated. The general conclusion to date is that laboratory tests are able to pick out an inferior grade of transformer oil. It is not expected that final decision as to the merits of laboratory tests in selecting the best grade of transformer oil and differentiating between that grade and a medium grade will be determined until several more years of operating experience have been obtained with these transformers.

Method D 117 – 43 has been revised and the new Tentative Method D 117 – 46 T has been accepted by the Society. This revision shows a reorganization of the methods in which D 117 – 46 T now contains only definitions, significance statements and reference to the proper test procedure for each individual characteristic of an insulating oil. Detailed methods of operation of the particular tests referred to have been removed from Standard Method D 117 and set up under their own A.S.T.M. designation.

A complete revision of the sampling procedure for insulating oil was prepared, to take the place of the old procedure described in Standard Method D 117 – 43. The final draft of the new sampling procedure accepted by Subcommittee IV has now been submitted to Committee D-9 for letter ballot with the intention that this procedure will ultimately appear as a separate standard under its individual number.[5]

Work during the year has indicated the necessity of changing the title of a method now appearing in Method D 117 – 43 as "free and corrosive sulfur" to "corrosive free sulfur," and to change the method to call for 7 hr. heating at 100 C. These revisions plus several others are ready for submission to Subcommittee IV as a new tentative method. It has also been agreed to conduct work on the development of a test for corrosive combined sulfur, and as soon as the test has been developed it will be submitted to Subcommittee IV for publication as a new tentative.

The membership of the subcommittee was circularized regarding the methods of test used for evaluating the oxidation stability of cable and capacitor oils. Wide variations in procedures are followed. The temperature range used by the different members varied between 85 and 150 C., and the duration of the aging between 24 hr. and 7 days. The tests performed to evaluate the end result are power factor, d-c. resistivity, and light transmission. Each of the members of the section has agreed to run a series of tests comparing their regular method with a standardized 96 hour aging at 100 C.

A new Tentative Method of Test for Power Factor and Dielectric Constant of Electrical Insulating Oils of Petroleum Origin[4] was prepared and submitted to the main committee for letter ballot. This method goes into detail concerning precautions that have to be taken to obtain checkable results when measuring power factor of insulating oils, but the main details concerning definitions, theory, measuring equipment and operating procedure will continue to be a part of the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 – 46 T).

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[5] See Editorial Note, p. 371.

Certain modifications have been made in the Tentative Methods of Test for Gas Content of Insulating Oils (D 831 – 45 T) and the revised method has been approved for adoption as standard.

A revised and greatly improved Method of test for Dielectric Strength of Insulating Oil of Petroleum Origin (D 877 – 46 T) was accepted by the Society as tentative. The subcommittee has voted to prepare a new Paragraph (*c*) to be added to the tentative method to permit taking five shots on a single cup of oil and averaging these five readings, as an alternate routine test. This proposal is being submitted to letter ballot of Subcommittee IV.

New statements on definition and significance have been accepted by Subcommittee IV on the tests for inorganic chlorides and sulfates, power factor and resistivity. These statements have been submitted to Committee D-9 for final approval. Work is being continued to develop statements on definition and significance of tests for saponification number, gas content of oil, and corrosive free sulfur and corrosive combined sulfur.

Work is continuing in an attempt to standardize a method which will accurately determine quantitatively the small amount of water that may be present in insulating oil. Work during the year has shown that refinements in the method have to be developed to prevent contamination by atmospheric moisture both during sampling and while testing the oil. Furthermore, retention of traces of moisture on the inside glass surface of the sample bottle has also been shown to produce inaccurate results. Work is being continued in an attempt to overcome these problems.

During the year the first round-robin test on interfacial tension was conducted. Samples of both new and used insulating oils were sent to members of the section studying this test and 14 out of 20 members completed their assignment. Differences as great as 14.0 dynes and 5.0 dynes were obtained on new and used oil respectively. It is believed that these differences are caused by one or more of the following conditions:

1. Failure to follow method submitted,
2. Poor quality of distilled water,
3. Improper cleaning of glassware,
4. Inadequate flaming of ring,
5. Improper standardization, and
6. Mechanical defects in tensiometer.

Work will be continued next year to try to eliminate the causes of the great variation in test results obtained to date.

The section working on the development of specifications for insulating oils has had two subsections attempting to obtain agreement on the screening specification referred to in the 1946 report.[6] These subsections have been attempting to prepare screening specifications on transformer oils, circuit breaker oils, and cable oils. Progress has been slow because of the great differences of opinion in the subcommittee and in the industry as to what characteristics and values should be included in this screening specification, but work is continuing with the hope that agreement may soon be reached.

Subcommittee IV sponsored a Symposium on Insulating Oils held during the past year at which the following papers were presented:

"Application of the Interfacial Tension Test in Grading Oil in Transformers Relative to Serviceability," by E. F. Walsh,

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[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 393 (1946).

"The Interfacial Tension Test and Its Significance in Appraising Performance of an Insulating Oil," by G. W. Gerell,

"Steam Emulsion Number as an Index of Transformer Oil Serviceability," by M. D. Baker, and

"Refresher on Statistical Analysis Applied to Two ASTM Oil Dielectric Strength Test Procedures," by E. W. Greenfield.

The first three papers were published in the May, 1947, issue of the A.S.T.M. Bulletin,[1] and the last paper by Mr. Greenfield will be published at a later date.[2]

A second symposium to be held in connection with the 1947 Annual Meeting, will comprise the following papers:

"The Advantages of an Inhibited Transformer Oil,"[2] by T. E. Reamer and R. G. Larsen,

"Oxidation Inhibitors in Electrical Insulating Oils,"[2] by Leo J. Berberich,

"Power Factor of Electrical Insulating Oils, Significance of, and Methods of Testing Stability,"[2] by J. C. Balsbaugh, and

"Serviceability Tests on Transformer Oil from the Viewpoint of the Maintenance Engineer,"[2] by Frank Pohnan.

Subcommittee V on Ceramic Products (K. G. Coutlee, chairman).—The sections of Subcommittee V have been reorganized as follows:

Section A on Stoneware and Similar Vitreous Products (K. G. Coutlee, chairman).

Section B on Glass and Glass Insulators (R. M. Havourd, chairman).

Section C on Steatite and other Special Purpose Ceramic Bodies (H. Thurnauer, chairman).

Section D on Glass Bonded Mica (L. J. Cavanaugh, chairman).

Further studies of the factors involved in the determination of the moisture sensitivity of power factor and dielectric constant of steatite are in progress.

Work has been started on the preparation of test methods for both compression-molded and injection-molded glass-bonded mica.

Subcommittee VI on Solid Filling and Treating Compounds (R. H. Titley, chairman) is gathering information preparatory to writing of specifications for solid filling compounds. A revision of Standard Methods D 176 is also under consideration. Statements on significance of test have been returned from Subcommittee XI and are being put into final form.

Subcommittee VII on Insulating Fabrics (R. W. Chadbourn, chairman).—During the year the subcommittee continued its study of the slow rate-of-rise dielectric strength test intended as a substitute for the existing step-by-step test for varnished cloth. The method was referred to the Insulated Power Cable Engineers' Association for comments, as the cable manufacturers make extensive use of the step-by-step test. This organization reported favorably on the method and, as no adverse criticisms have been received to date, the method will be written up for final review. If accepted, the method will be referred to Subcommittee XII with the recommendation that it be incorporated in Standard Methods D 149 as an alternate to the step-by-step method.

A limited amount of additional data was accumulated on dielectric breakdowns of tapes subjected to 12 per cent elongation.

The subcommittee is studying the problem of tests at high humidities on varnished cloth to simulate conditions of use in tropical areas where the humidity may be very high for prolonged periods. Either 90 or 96 per cent relative humidity will probably be adopted as a standard atmosphere for such tests.

The statements of significance of tests for varnished cloth were slightly revised as the result of comments from Subcommittee XI, and are being reconsidered by Subcommittee VII. Statements of significance of tests for treated sleeving are now being reviewed by the subcommittee.

The Section on Treated Sleeving is

contemplating adoption of wall thicknesses for treated sleeving as soon as the manufacturing situation becomes sufficiently stabilized.

During the year, the test procedures for glass fabrics were divorced from Tentative Methods D 295 and issued in the form of Tentative Method of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used in Electrical Insulation (D 902 - 46 T). The section is now engaged in the development of a suitable endurance test.

The Section on Electrical Adhesive Tapes has virtually finished development of tests for thickness, adhesivity, breaking strength, and shelf ageing (heat and moisture). Two methods of test for electrolytic corrosion are being studied, and also a method for metal corrosion. The method for effect of varnishes and solvents on tapes is receiving further consideration.

Subcommittee VIII on Insulating Paper (C. T. Hatcher, chairman).—The section which has been working with members of Committee D-6 on Paper and Paper Products completed a method to be used for pH determination of insulating papers. This method, with approval of Committee D-9, is being issued. The joint D-9 - D-6 Group is working on a conductivity method for aqueous extract of paper. Round-robin tests are being conducted in order to determine if the proposed method is satisfactory. Requests were received for a pH method for vulcanized fiber. This subject is being considered by the section.

Consideration is being given to the replacement of the present test for acidity and alkalinity in Method D 202 with an up-to-date electrometric method.

A sampling method and air resistance method was approved by Committee D-9. With the approval of these two methods, the work of reviewing the methods for making physical tests on insulating papers so as to bring them into agreement with D-6 methods whenever possible was completed.

The revised tensile breaking strength method given in the Appendix, was prepared by Subcommittee VIII.

Several requests were received relative to the preparation of purchase specifications for insulating papers. A survey in Committee D-9 indicates that there is a need for such specifications, and a new section has been formed to study and prepare specifications covering the purchase of insulating paper.

Subcommittee IX on Mica products (E. O. Hausmann, chairman).—Last year Subcommittee IX combined the Standard Methods of Tests for Grading and Classification of Natural Mica, (D 351 - 38) and the Tentative Method of Test for Power Factor and Dielectric Constant of Natural Mica (D 351 - 42 T), into the present Standard Methods of Testing, Grading and Classifying Natural Mica (D 351 - 46). In this standard there is a method for classifying the visual quality of block mica and mica splittings. Seven classes of mica are defined in this general classification. Since this is a visual method, the committee has asked the Mica Fabricators' Association to submit seven samples of mica which could be considered as typical of the seven classes of material given in the method. These seven samples have been accepted by Committee D-9 as standards for this classification.

It is contemplated that colored photographic transparencies will be made of these samples, which transparencies will then be made available for distribution and use as secondary standards.

The Specifications for Natural Block Mica and Film Suitable for Use in Fixed Mica-Dielectric Capacitors (D 709 - 46 T) have been in use for a short period of time and have been subjected to some criticism, largely because the suppliers were not familiar with the methods used for evaluating the qualities of these materials in accordance with these specifications. However, considerable progress has been made in the process of education which is necessary to make these specifications useful and acceptable. Some minor revisions of these specifications have been prepared and are under consideration for adoption.

Subcommittee X on Conditioning (M. L. Macht, chairman).—A revision of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 - 46 T) which includes a standard laboratory atmosphere, a standard room temperature, ten standard test temperatures, and three procedures for conditioning prior to test has been voted on favorably by letter ballot of Committees D-9 and D-20. Further revisions which will include additional test temperatures and procedures for conditioning prior to test are now in course of preparation.

Subcommittee XI on Significance of Tests (J. H. Adams, chairman).—During the year there has been a gradual but gratifying increase in interest in the work of Subcommittee XI. Twenty-three are listed as members of whom most have been active. As a reviewing body, it is very desirable that the membership be versed collectively in a very wide variety of tests and materials.

Statements of test significance which Subcommittees VI and VII proposed to add to test methods under their jurisdiction were reviewed and recommendations made. The efforts of the committee were directed primarily to establishing a Recommended Practice for Writing Statements as to the Usefulness of Tests of Electrical Insulating Materials which would establish the desired content of such statements. It is expected that this will soon be accomplished. Information was exchanged with Subcommittee X on Definitions, Nomenclature, and Significance of Tests, of Committee D-20, and an inquiry was circulated as to the possibility of establishing some common policy for the Society regarding statements of test significance or utility.

Subcommittee XII on Electrical Tests (K. N. Mathes, chairman).—In Section A of this subcommittee, a review of the resistivity methods is under way. Some revisions of the present standard procedures are being considered and, no doubt, these will be presented at the next meeting of the subcommittee.

Section C on Power Factor Measurements is considering methods for measuring power factor frequency above those which have been standardized in the present procedures. These frequencies range from 50 to 10,000 megacycles per second.

The Section on Arc Resistance has been very actively working on methods used for evaluating the arc resistance of insulating materials. The present method is far from being satisfactory and further changes are under consideration. This seems to be a very elusive problem and the section working on it has spent many hours in trying to find a satisfactory method for arc-resistance measurements.

Subcommittee XIII on Mechanical Tests (C. R. Stock, chairman).—During the past year, Subcommittee XIII has been mainly active in reviewing two types of mechancal tests as they apply to electrical insulation. Section A has reviewed the situation existing among various A.S.T.M. test methods for performing compression tests, and is working at present on a generalized method which is intended to serve as a reference for tests for specific materials. Section B has distributed questionnaires regarding the relative utility of tension tests for plastics, Method D 651 *versus*

Method D 638. The result of this questionnaire was to show that both are useful. Consideration is also being given to the writing of a preferred procedure for specifying tension tests, intended to point out the necessary factors to be covered in performing tension tests on any electrical insulation, including also such materials as papers and tapes.

This report has been submitted to letter ballot of the committee which consists of 133 voting members; 68 members returned their ballot, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. W. ORR,
Chairman
GORDON THOMPSON,
Vice-Chairman

W. A. ZINZOW,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Natural Block Mica and Mica Films Suitable for Use in Fixed Mica-Dielectric Capacitors (D 748 - 45 T), and
Laminated Thermosetting Materials (D 709 - 46 T).

Revision of Tentative Methods of:

Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 - 46 T),
Testing Electrical Insulating Oils (D 117 - 46 T),
Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 - 46 T),
Measuring Dimensions of Rigid Rods Used in Electrical Insulation (D 741 - 43 T),
Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used in Electrical Insulation (D 902 - 47 T),
Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 - 46 T).

Tentative Revision of Standard Methods of:

Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 46), and
Measuring Dimensions of Rigid Tubes Used in Electrical Insulation (D 668 - 44).

These recommendations were accepted by the Standards Committee on September 4, 1947, and the revised tentatives and tentative revisions appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON ELECTRICAL INSULATING MATERIALS

In this Appendix are given recommendations affecting certain standards and tentatives covering electrical insulating materials which are referred to earlier in this report.[1] The standards appear in their present form in the 1946 Book of A.S.T.M. Standards, Part III-B.

NEW TENTATIVES

Tentative Specifications for Nonrigid Polyvinyl Tubing:

These specifications are intended for use in procurement of Nonrigid Polyvinyl Tubing used almost exclusively for electrical insulation purposes. They have been prepared by a special section of Subcommittee III of Committee D-9 constituted in accordance with the rules and By-Laws of the Society. It is recommended that these specifications[2] be accepted for publication as tentative.

Tentative Method for Sampling Electrical Insulating Oils:

This method was formerly a part of Standard Methods of Testing Electrical Insulating Oils (D 117 - 43). A new tentative form of this method has now been issued under the designation D 117 - 46 T, which consists of a series of references to several methods of testing, in which particular methods are described in detail. It is recommended that this method[2] be accepted for publication as tentative, and after acceptance that a reference to it be added in Methods D 117 - 46 T.

Tentative Method of Test for Power Factor and Dielectric Constant of Electrical Insulating Oil of Petroleum Origin:

This is another of the methods formerly included in Standard Methods of Testing Electrical Insulating Oils (D 117 - 43). It is recommended that this method[2] be accepted for publication as tentative, and after acceptance that a reference to it be added in Methods D 117 - 46 T.

REVISIONS OF TENTATIVES

Tentative Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 46 T):

This revision concerns the procedure to be used in tensile strength testing of paper. It deletes from this method a description of test procedures formerly used and refers instead to the procedure used for tension testing of paper as described in the more generally applicable method in the Tentative Method of Test for Tensile Breaking Strength of Paper and Paper Products (D 828 - 45 T), which method is under the jurisdiction of Committee D-6. It is a part of a general program of simplification and unification of test methods applicable to the same types of materials. It is accordingly recommended that

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[1] See p. 364.

[2] These methods and specifications were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B.

Sections 10 to 13, inclusive, of Methods D 202 be replaced by the following new section:

10. *Tensile Strength Procedure.*—Tensile strength shall be determined in accordance with the Tentative Method of Test for Tensile Breaking Strength of Paper and Paper Products (A.S.T.M. Designation D 828), except that the material shall be sampled and conditioned in accordance with Sections 2 and 3 of these methods.

Since the best method for determination of air resistance is given in the Tentative Method of Test for Resistance of Paper to Passage of Air (D 726 – 46 T), Committee D-9 has decided to delete the present Sections 24, 25, and 26 and replace them by a new section. It is accordingly recommended that these sections be deleted and replaced by the following new section:

21. *Procedure.*—Air resistance shall be determined in accordance with the Tentative Method of Test for Resistance of Paper to Passage of Air (A.S.T.M. Designation: D 726), except that the material shall be sampled and conditioned in accordance with Sections 2 and 3 of these Methods D 202.

NOTE.—Method D 726 provides for use of a rubber gasket to prevent leakage over the surface of the test specimen. This refinement may give substantially higher test results than would be obtained on the ungasketed air resistance apparatus.

Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 – 43 T):

The purpose of this revision is to introduce a standardized notching procedure in the preparation of specimens for the impact test. This revision, with an editorial modification suggested by Committee D-9, has been accepted and is jointly recommended for acceptance by Committees D-9 and D-20.

Section 3 (a).—Change the second sentence of this paragraph to read as follows: "To insure the correct contour and condition of the specified notch, all specimens shall be notched in accordance with the directions in Section 4."

New Section.—Add a new Section 4 to read as follows, renumbering the subsequent sections accordingly:

4. *Notching Test Specimens:* (*a*) Notching shall be done with a milling cutter. The cutting edge shall be carefully ground and honed to insure sharpness and freedom from nicks and burrs. The included cutting angle of the tool shall be 45 ± 1 deg. with a radius of 0.010 ± 0.001 in. at the intersection of the arms of this angle. Tools with no rake and with a work-relief angle of 15 to 20 deg. have been found satisfactory. For those materials for which it has been established that wider tolerances on the notch contour and dimensions will not affect the test results significantly, the wider tolerances may be used.

(*b*) The tool shall be so mounted that the plane which bisects the included cutting angle is perpendicular to the axis of rotation within plus or minus 2 deg. Any standard milling machine or lathe may be used for the cutting operation.

(*c*) Inspection of a notched specimen may be made instead of inspection of the cutter for any material in which the relation between notch dimensions and cutter dimensions has been established. The contour and alignment of multi-tooth cutters shall be determined from the notch which the cutter produces in a suitable material.

(*d*) The linear speed of the cutting tool shall not exceed 600 ft. per min. (Note 1).

(*e*) The feed shall be slow enough to result in a smooth cut. Speeds in the range from 0.5 to 5.0 in. per min. have proved satisfactory for some materials (Note 1).

NOTE 1.—While the speed ranges given in Paragraphs (*d*) and (*e*) have produced satisfactory notches in some plastic and electrical insulating materials, it may be necessary to study the effect of variation in these conditions when unfamiliar materials are to be notched.

(*f*) After each 500 notches, or more often, if hard abrasive materials are being notched, the cutter or a notched specimen shall be inspected (Note 2) for sharpness, freedom from nicks, radius of tip and angle. If any signs of dullness are apparent, or if the angle and radius do not fall within their specified limits the cutter shall be replaced with a newly sharpened and honed one. A microscope with a camera lucida attachment is suitable for checking the radius and angle of either the cutter or notch.

NOTE 2.—Relatively close tolerances must be imposed upon the angle and radius of the notch for most materials, because these factors largely determine the degree of stress concentration at the base of the notch during the test. The maintenance of a sharp, clean-edged cutting tool is particularly important since minor defects at the base of the notch can cause large errors in test results.

Section 8 (a).—Change the second sentence to read as follows: "To insure the correct contour and condition of the specified notch, all specimens shall be notched as directed in Section 4."

Tentative Specifications for Flexible Treated Cotton and Rayon Sleeving Used in Electrical Insulation (D 372 - 45 T):

These revisions provide for the substitution of three subgrades of sleeving for Grade C in the present specifications. This is being done to make available for use commonly manufactured and used materials within the limits of these specifications. These revisions are all concerned with the requirements of the three subgrades of material being added to the specifications.

Section 1.—Replace the paragraph on Grade C with following:

Grade C-1.—A braided cotton or rayon sleeving impregnated with an oil varnish. It shall have a maximum flexibility It is generally known as "triple saturated sleeving," and has a lower dielectric strength than grades B-1 or B-2. This grade shall conform to all requirements of these specifications, including the heat-endurance test (Section 6).

Grade C-2.—A treated sleeving having a construction similar to grade C-1. It is generally known as "double saturated sleeving," and has a lower dielectric strength than grade C-1. This grade shall conform to all requirements of these specifications, including the heat-endurance test (Section 6).

Grade C-3.—A treated sleeving having a construction similar to grade C-1. It is generally known as "single saturated sleeving." This grade shall conform to the requirements of these specifications, except the aging test (Section 5), the heat-endurance test, (Section 6), and the rate of burning test (Section 7).

Section 4 (c).—Change to read as follows:

(*c*) Grade C treated sleeving shall show the following values with the short-time dielectric strength test:

Grades	*Average*	*Minimum*
C-1	2500	1500
C-2	1500	800
C-3	No dielectric values guaranteed	

No one of the ten puncturing voltages shall be less than the minimum values listed for each grade.

Section 5.—Change the next to last line of this section to read as follows:

Grades A-1, A-2, C-1, and C-2	96 hr
Grades B-1 and B-2	72 hr

Section 6.—Change to read as follows:

6. When tested for heat endurance in accordance with Section 17 of the A.S.T.M Standard Methods D 350, Grades A-1, B-1, C-1, and C-2 treated sleeving shall neither soften, blister, nor flow.

Section 7.—Change to read as follows:

7. All grades of treated sleeving, except Grade C-3, furnished under these specifications shall require at least 45 sec. to burn a length of 1 in. (2.54 cm.).

Tentative Specifications for Vulcanized Fibre Sheets, Rods and Tubes Used for Electrical Insulation (D 710 - 43 T):

This revision involves a rather minor change in one of the sentences used to describe the characteristics of "Bone Grade," one of three classes of materials covered by these specifications.

Section 3.—Change the last sentence under "Bone Grade," from its present form, namely:

It possesses maximum hardness and physical strength as well as excellent turning, threading and milling characteristics.

to read as follows:

It possesses maximum hardness and physical strength as well as excellent machining and electrical characteristics.

Tentative Method of Test for Rockwell Hardness of Plastics and Electrical Insulating Materials (D 785 – 44 T):

This revision concerns changes in procedure and scale designations which have been arrived at as the result of a series of investigations concerning reasons for interlaboratory variations in test results. Some time intervals, and consequent test results, have been changed. It is generally recognized that test results are dependent on the time intervals used. In the interest of uniformity of test results the time intervals involved have been changed to coincide with less critical values.

Section 1.—Add the following as a new Paragraph (*c*):

The results obtained by this method are not generally considered as a measure of the abrasion or wear resistance of the plastic.

Section 2.—Add the following as a new Paragraph (*b*):

(*b*) For Rockwell hardness testing, it is necessary that the major load be completely supported by the specimen, when the major load is fully applied, and not be held by other limiting elements of the machine. To determine whether this condition is satisfied the major load should be applied to the test specimen. If an additional load is then applied, by means of hand pressure on the weights, the needle should indicate an additional indentation. If this is not indicated the major load is not being applied to the specimen and a long stroke (PL) machine or other scale combination should be used. For the harder materials a stroke equivalent to 150 scale divisions, under major load application, may be adequate, but for softer materials the long stroke (250 scale divisions under major load) machine is required.

Section 5 (*c*).—Change to read as follows:

(*c*) Unless otherwise specified in the individual methods or specifications for a particular material, either the R, L, M, or E scale (Note 1) shall be used. The choice of scales to be used shall be governed by the considerations concerned with total indentation readings and the final scale reading for a particular material and scale.

(1) If the total indentation, reading with major load applied, for a particular scale exceeds the limits of the test machine used (150 divisions for regular machines and 250 divisions for PL machines) the next less severe scale shall be used. Thus, if the M scale indentation (with major load) is 290 divisions the L scale shall be used.

(2) If the final scale reading is high the method will not give a good spread in values and therefore will not adequately differentiate the harder materials. Therefore, it is suggested that if the final reading for any particular load and indenter combination exceeds 115 that the next harder scale be used. For example, if the R scale value exceeds 115 the L scale may be used. The total indentation shall be determined by the number of divisions the pointer passes through during 15 sec. from the time of tripping the handle.

Section 5 (*e*).—Change to read as follows:

(*e*) With the specimen in place, the minor load of 10 kg. shall be applied and the zero setting made within 10 sec.; the major load shall be applied *immediately* after the zero setting has been completed. The major load shall be removed from the specimen 15 sec. after the handle has been tripped, the time being determined by a stop watch. The Rockwell readings on the red scale shall be taken to the maximum nearest full scale division reached 15 sec. after removing the major load.

Notes 1 and 2.—Delete these two notes, substitute the following as a new Note 1 and renumber the present Note 3 as 2:

NOTE 1.—The scale requirements, using the red scale, are as follows:

Scale	Minor Load, kg.	Major Load, kg.	Penetrator
R......	10	60	½-in. ball
L......	10	60	¼-in. ball
M......	10	100	¼-in. ball
E......	10	100	⅛-in. ball

Tentative Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used in Electrical Insulation (D 902 – 46 T):

The following revisions recommended in these methods are of two types; first, clarifying changes resulting from the first examination of the new tentative after publication and second, a change in requirements for test temperatures to be used for silicone-coated fabrics:

Section 2.—Change title of this section from "Selecting Rolls" to read "Selecting Sample Rolls."

Section 3 (a).—Delete the second and third sentences as the individual test methods describe these requirements.

New Section.—Add a new Section 4 to read as follows, renumbering the subsequent sections accordingly:

> 4. *Selecting Test Specimens.*—The test specimens shall be prepared from samples as selected in Section 3 and as provided for in the individual test methods.

Section 4.—Change the word "samples" to read "specimens" wherever it occurs.

Section 15 (b).—Change to read as follows:

> (*b*) Specimens cut from the full width cloth samples or from sample rolls of tapes over 1 in. (2.54 cm.) in width shall be 1 in. (2.54 cm.) in width and tested between electrodes specified in Section 14.

Section 18.—Add a new paragraph (*d*) to read as follows:

> (*d*) In the case of the short-time test, ten punctures shall be made on each specimen, while in the case of the step-by-step test, five punctures shall be made.

Section 22.—Delete the second sentence and change the third sentence to read as follows: "The electrode pressure on the specimen shall be not less than 10 nor more than 20 psi."

New Section.—Add a new Section 23 to read as follows, renumbering the subsequent sections accordingly:

> 23. *Test Specimens.*—Each specimen shall be of such size that it shall extend beyond the guard electrode for a distance of at least four times the thickness of the specimens.

Present Section 24 (b).—Change to read as follows: "Three specimens shall be tested in single thicknesses at each temperature."

TENTATIVE REVISION OF STANDARD

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulating Materials (D 229 - 46):

This revision is a modification of a tentative revision of this standard originally submitted in June, 1943, and modified in August, 1943, and June, 1944. This is a limitation on the thickness of the specimens to be tested by a revised procedure.

Sections 7 to 10.—Change the italic heading preceding these sections to read "For Materials Under $\frac{1}{8}$ in. in Thickness," retaining the present footnote 5.

New Section.—Add a new Section 11 to read as follows, renumbering the subsequent sections accordingly:

> *For Materials $\frac{1}{8}$ in. or Over in Thickness*
>
> 11. (*a*) Except for definitions of cutting directions of specimens as specified in Section 2 and for conditioning as specified in Section 3, the flexural strength properties including modulus of rupture and modulus of elasticity in bending shall be determined in accordance with the Tentative Method of Flexural Test of Plastics (A.S.T.M. Designation: D 790).[a]
>
> (*b*) Five specimens cut lengthwise and five specimens cut crosswise of the sheet shall be tested for flatwise test. Where edgewise tests are desired, five specimens shall also be cut lengthwise and five crosswise of the sheet.

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[a] 1946 Book of A.S.T.M. Standards, Part III-B.

EDITORIAL CHANGE IN STANDARD

Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (D 392 - 38):

Figure 1.—Change the top dimension of the funnel from "$3\frac{3}{16}$" to read "$3\frac{21}{32}$" in order to agree with the original 40 deg. angle. Also show 40 deg. for the angle of the funnel.

REPORT OF COMMITTEE D-10*

ON

SHIPPING CONTAINERS

Committee D-10 on Shipping Containers held one meeting during the year at Philadelphia, Pa., on April 10–11, 1947, with 48 members and visitors in attendance. Visitors included one representative from the Imperial Chemical Industries, Cheshire, England; one from the Transportation Corps Board, New York Port of Embarkation; and five from industrial concerns, an indication of the continued interest in shipping container test methods.

The committee voted to continue its Spring Meeting at the time of the American Management Association Packaging Show, but recommended more time for its deliberations and subcommittee meetings.

The committee was fortunate to inaugurate a new service to the membership through one of its own members, Julian H. Toulouse. At the Spring Meeting Mr. Toulouse presented a sound motion picture in color and an illustrated lecture on "A Study of Shipping Container Performance."

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1946 Annual Meeting, Committee D-10 presented to the Society through the Administrative Committee on Standards the proposed Tentative Method of Test for Water-Vapor Permeability of Packages. On subsequent recommendation of Committee D-10 publication of this method as tentative was withheld. On May 7, 1947, the Standards Committee accepted the further recommendation of Committee D-10 that the method with certain editorial changes be published as tentative. It has been assigned the designation D 895 – 47 T, and will appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B, and has been issued separately as a reprint.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following four tentatives, with revisions in three methods as indicated, be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Method of Drop Test for Shipping Containers (D 775 – 45 T),[1] without revision,

Tentative Method of Compression Test for Shipping Containers (D 642 – 45 T),[1] revised as follows:

Section 3 (a).—Delete the first sentence and substitute the following two sentences: "The containers being tested shall be complete in all respects. Depending on the purpose of the test, interior packing may or may not be included."

Section 4 (b).—Delete the fourth sentence which reads: "The adhesive shall be kept at least $\frac{1}{2}$ in. away from the score lines."

Tentative Method of Test for Shipping Containers in Revolving Hexagonal

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] 1946 Book of A.S.T.M. Standards, Part III-B.

Drum (D 782 - 46 T),[1] revised as follows:

Section 6.—At the end of this section, delete Item *(4)* which reads: "The contents have been damaged."

Tentative Method of Incline Impact Test for Shipping Containers (D 880 - 46 T),[1] revised as follows:

Section 2 (a).—Delete the fifth sentence which reads: "(For tests of fiberboard containers the face of the dolly shall be covered with canvas.)"

The recommendations appearing in this report have been submitted to letter ballot[2] of the committee, the results of which will be reported at the Annual Meeting.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definitions of Terms (Edward Dahill, chairman) is preparing a new draft of definitions of terms, which had been previously submitted to the committee but was referred back to the subcommittee for further study.

Subcommittee II on Methods of Testing (E. R. Stivers, chairman) is continuing a study of a vibration test method and test methods covering bags, sacks, and cylindrical containers. It is also gathering information on typical failures of large crates and boxes so that it can suggest a test method for such containers.

Subcommittee III on Moisture and Water-Vapor Resistance (R. W. Lahey, chairman) has prepared a new draft of a proposed Tentative Method of Test for Water Resistance of Containers, Spray Method, which had been previously submitted to the committee but was referred back to the subcommittee for further study. The subcommittee is also preparing drafts for a proposed tentative Method of Test for Water Vapor Permeability of Shipping Containers and Water Resistance of Containers, Submersion Method.

Subcommittee IV on Performance Standards (J. H. Toulouse, chairman) has started work on performance standards and can be expected to make suggestions before long.

Subcommittee VI on Interior Packing (W. B. Lincoln, Jr., chairman) has made some progress on standards for interior packing and is continuing study of this subject.

This report has been submitted to letter ballot of the committee, which consists of 69 members; 60 members returned their ballots, of whom 59 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. A. CARLSON,
Chairman.

EARL R. STIVERS,
Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-10 presented to the Society through the Administrative Committee on Standards the proposed Tentative Method of Test for Water Resistance of Containers by the Spray Method. The new tentative method was accepted by the Standards Committee on November 6, 1947, and appears in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B, under the designation D 951 - 47 T.

REPORT OF COMMITTEE D-11*

ON

RUBBER AND RUBBER-LIKE MATERIALS

Committee D-11 on Rubber and Rubber-Like Materials held one meeting during the year in Philadelphia, Pa., on February 27, 1947, in connection with the spring group meetings of A.S.T.M. committees. Several of the subcommittees have met separately during the year and considerable work has been carried on by correspondence. At the spring meeting, the committee voted to sponsor a Symposium on Rubber Testing to be given at the 1947 annual meeting. This symposium consists of eight papers which summarize many of the developments in rubber testing which have taken place during the war years, both in connection with the natural rubber and the synthetic rubber program.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1946 Annual meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards the following recommendations for revision of tentative specifications and methods. These were accepted by the Standards Committee on September 9, 1946, and the revised tentatives appear in the 1946 Book of A.S.T.M. Standards, Part III-B.

Revision of Tentative Specifications for:

Rubber Sheath Compound for Electrical Insulated Cords and Cables (D 532 - 39 T),

Insulated Wire and Cable: Ozone-Resistant Type Insulation (D 574 - 40 T),

Insulated Wire and Cable: Heat-Resisting Synthetic Rubber Compound (D 754 - 43 T),

Insulated Wire and Cable: Performance Synthetic Rubber Compound (D 755 - 44 T), and

Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 - 46 T) (Two separate revisions).

Revision of Tentative Methods of:

Testing Rubber Insulated Wire and Cable (D 470 - 46 T),

Testing Rubber Hose (D 380 - 40 T),

Testing Rubber-Coated Fabrics (D 751 - 43 T), and

Identification and Quantitative Analysis of Synthetic Elastomers (D 833 - 45 T).

The revision of the Tentative Specifications for Rubber Sheath Compound for Electrical Insulated Cords and Cables (D 532) deleted the requirements for vulcanization in a metal mold which is considered superfluous if the physical property requirements of the rubber are met. In the Specifications for Ozone Resistant Insulation (D 574) the value of the constant *K* in the formula for insulation resistance was changed from 5,280 to 2,000. During the war this constant was dropped to 1,000, and it is now felt that 2,000 is satisfactory when the emergency provisions are not used. The revision of the Tentative Specifications for Heat Resisting Synthetic Rubber Compound (D 754) permits the use of some natural rubber with synthetic rubber as it becomes available and also changes Table II on thickness of insulation to include No. 12 and 14 A.w.g. conductors with $\frac{1}{32}$-in. wall. In the Tentative Specifications for Performance Synthetic Rubber Compounds (D 755) the same changes were made as in Specifications D 754 and also, aging requirement after oxygen bomb aging was made more severe. The revision of Tentative Specifications for Class AO Rubber Compound (D 27) covers a change in Table II on thickness of insulation to include No. 12 and 14 A.w.g. with $\frac{1}{32}$-in. wall for 0 to 600 v. in addition to the present values for $\frac{3}{64}$-in. wall. It also provides that on these sizes the $\frac{1}{32}$-in. insulation will be furnished, unless otherwise specified. In addition, Specifications D 27 were revised to include the use of code braids and the horizontal flame test which previously appeared as emergency alternate provisions.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

The revision of the Tentative Methods of Test for Insulated Wire (D 470) adds the horizontal flame test developed during the war and previously published as an emergency alternate provision. In the Tentative Methods of Testing Rubber Hose (D 380) the section on test specimens for the tensile strength test previously contained a maximum requirement on thickness of $\frac{1}{8}$ in. and no minimum. Since specimens buffed thinner than $\frac{1}{32}$ in. do not give reliable results, this thickness was added as a minimum requirement together with a provision for using certified test slabs of proper thickness prepared from the same material as used in the hose when specimens of the required thickness cannot be obtained. The Tentative Methods of Testing Rubber-Coated Fabrics (D 751) were changed by having the sections dealing with apparatus and procedure for the hydrostatic resistance test completely rewritten including the addition of a new alternative method particularly applicable to fabrics having low resistance to the passage of moisture. The Tentative Methods of Identification and Quantitative Analysis of Synthetic Elastomers (D 833) were extensively revised to include new methods of identification of synthetics by means of spot tests in place of those previously given. The new methods are more rapid and fully as reliable as the older ones and represent utilization of the rapid advances which are being made in the synthetic rubber field.

Many of the above mentioned revisions pertaining to insulated wire were made in order to permit the elimination of emergency alternate provisions previously published. Accordingly, the Emergency Alternate Provisions EA-D 27a, EA-D 574b, EA-D 754, EA-D 755, and EA-D 470 were withdrawn by recommendation of Committee D-11 and acceptance of the recommendation by the Standards Committee on September 9, 1946.

I. NEW TENTATIVES

Tentative Methods of Test for Contact and Migration Stain of Vulcanized Rubber in Contact with Organic Finishes.[1]—These methods of test give procedures for producing under standardized conditions stains caused by vulcanized rubber compounds on organic finishes. Some rubber compounds when held continuously in contact with or adjacent to light-colored finishes and exposed to conditions of heat, pressure or sunlight produce objectionable discoloration of the finish. This is particularly true in the case of such assemblies as white refrigerators or light-colored automobile bodies. A standard means of evaluating such staining characteristics is urgently needed and has been given considerable study in Technical Committee A on Automotive Rubber which has developed this proposed method. The method does not propose a standard finish for use in the test but instead requires that the test be performed against the finish actually involved in the service application being investigated. The evaluation of the degree of stain is accomplished by visual comparison with unstained specimens.

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[1] These methods were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B.

Tentative Method of Test for Plasticity and Recovery of Rubber and Rubber Like Materials by Means of the Parallel Plate Plastometer.[1]—This tentative method has been prepared by Subcommittee XXVI on Processibility Tests and gives the procedure for determining the plasticity and recovery of uncompounded, compounded or reclaim stocks of unvulcanized rubber and rubber-like materials. The method employs the Williams parallel plate plastometer which has been used for a number of years in connection with the evaluation of the processing characteristics of rubber compounds. Many variations in test conditions have been used and a standardized procedure will be of considerable value.

Tentative Method of Test for Viscosity by Means of the Shearing Disk Viscometer.[1]—This tentative method has also been prepared by Subcommittee XXVI on Processibility Tests and covers a standard procedure for determining the viscosity of uncompounded, compounded or reclaim stocks of unvulcanized rubber and rubber-like materials by means of the Mooney viscometer. The Mooney viscometer has been very widely used during the war and has furnished the principal basis for the control of processibility of synthetic rubber produced in the government program. This standardized procedure for use of the viscometer takes into consideration the experience obtained in use of the equipment during the war program and should be of definite value in the rubber industry. In addition to the use of the method in processibility measurement, it affords a means for evaluation of rate of cure and scorching characteristics of compounded stocks. The committee may at a later date wish to revise the method to cover these additional uses or it may wish to recommend additional tentatives dealing with these phases but it seems advisable to make the present method available at as early a date as possible.

II. REVISIONS OF TENTATIVES

The committee recommends that the following three tentatives be revised as indicated below and continued as tentatives:

Tentative Methods of Test for Compression Set of Vulcanized Rubber (**D 395 - 46 T**):[2]

Section 5.—Add the following paragraph:

> When the standard specimen ½ in. in thickness cannot be obtained from the rubber sample or article being tested, it is permissible to assemble a specimen having the required thickness by plying together, without cementing, thinner layers of approximately equal thickness. Such plies shall be smooth, of uniform thickness and shall not exceed four in number. The resulting assembly shall conform to the outside diameter and total thickness specified for the standard specimen.

Subcommittee XVII has carried out an extensive series of tests to ascertain whether the use of plied specimens would seriously affect the results of the compression set test and has concluded that four plies may be used without serious consequences in assembling the test specimens of the standard thickness. Full account of this work is given in the Appendix to this report.[3] Since it very frequently happens that compression-set tests are desired on material which is not available in thicknesses sufficient for the standard specimen, prompt publication of this provision to give greater flexibility in testing is very desirable.

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[2] 1946 Book of A.S.T.M. Standards, Part III-B.
[3] See p. 387.

Tentative Method of Test for Indentation of Rubber by Means of the Durometer (**D 676 – 46 T**).[2]—Change the title by substituting the article "a" for "the" before the word "durometer" and replace the text with the revised procedure as appended[4] to this report. This tentative method has been completely rewritten in order to permit the use of the Rex durometer or any other complying instrument, as well as the Shore durometer. These instruments differ slightly from each other in some details but the hardness numbers are according to the same scale and the instruments are widely used interchangeably. As previously worded, the method did not permit the use of a maximum reading type of durometer. In revising the method, the calibration procedure has been clarified and some errors corrected.

Tentative Method of Heat Aging of Vulcanized Natural or Synthetic Rubber by Test Tube Method (**D 865 – 46 T**).[2]—As originally adopted, this method provided for the use of stoppered test tubes which limited the available air supply in contact with the test specimens. Subsequent work by the committee has shown that better reproducibility is obtained when provision is made for the entrance of additional air into the test tubes by convection currents through tubes inserted in the stoppers. A separate account of this work will be published in the A.S.T.M. BULLETIN, August, 1947.[5] The committee therefore recommends that the following changes be made in the Tentative Method D 865:

Section 1.—In first sentence, change ". . .to high-temperature aging in a controlled and limited quantity of air" to read, "to high-temperature aging under controlled conditions of air circulation."

Section 2(a).—Change the second sentence to read as follows: "In this method the test involves exposure of specimens to circulating air at an elevated temperature and at atmospheric pressure."

Section 4(b).—Add the following sentence to the end of this paragraph: "A suggested means for holding the specimens is shown in Fig. 1."

New Figure.—Add the accompanying Fig. 1 as a new Fig. 1.

Section 4(c).—Change to read as follows: "The test tubes shall be provided with a No. 20 cork stopper through which an inlet and an outlet tube shall be placed as shown in Fig. 1."

New Paragraph.—Add the following as a new Paragraph (*g*): "Means shall be provided to remove oil fumes from the oil bath so that they will not enter the inlet tube."

Section 8(a).— In the first sentence change "stoppered test tubes" to "test tube assembly."

Section 8(c).—Change to read as follows: "The test tube and the circulation tubes shall be carefully cleaned following each test or prior to each new test to remove any deposits of volatile material that may have condensed on the sides of the test tube or in the circulation tubes."

III. ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for GR-M Polychloroprene Sheath Compound for Electrical Insulated Cords and Cables Where Extreme Abrasion Resistance is Not Required (D 753 – 44 T),[2] and

Tentative Method of Test for Hydrogen Permeability of Rubber-Coated Fabrics (D 815 – 44 T).[2]

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[4] These revised methods were accepted by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B.

[5] **A. E. Juve, H. Boxser, D. F. Reahard, Jr., S. R. Doner, J. F. McWhorter, C. G. Cashion, G. D. McCarthy and R. M. Howlett, "The Effect of Air Circulation by Convection in the Test Tube Method of High Temperature Aging," ASTM BULLETIN, No. 147, August, 1947.**

Both of these two tentatives have been published for several years and are in regular use. No criticisms or suggestions for improvement have been received by the committee.

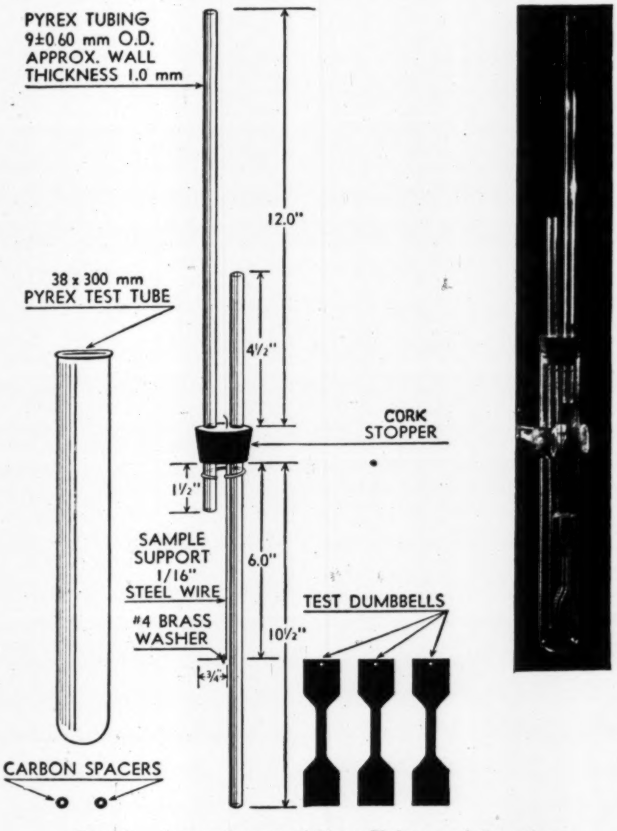
PYREX TUBING
9±0.60 mm O.D.
APPROX. WALL
THICKNESS 1.0 mm
12.0"
38 x 300 mm
PYREX TEST TUBE
4½"
CORK
STOPPER
1½"
SAMPLE
SUPPORT
1/16"
STEEL WIRE
6.0"
#4 BRASS
WASHER
10½"
¾"
TEST DUMBBELLS
CARBON SPACERS

Fig. 1.—Arrangement of Vent Tubes and Samples.

IV. Revision of Standard, Immediate Adoption

The committee recommends for immediate adoption revision in the Standard Methods of Test for Abrasion Resistance of Rubber Compounds (D 394 – 46) whereby Sections 12, 13, and 14 under "Method C. United States Rubber Co. Abrader" are deleted from the standard. A survey with respect to this Method C indicated that a total of fifteen machines of the type required had been manufactured and sold. When contacted, twelve purchasers replied to the committee's questionnaire. Seven of these voted to delete the method, three were noncommittal, and two voted for its retention. A subsequent letter ballot in the subcommittee yielded seventeen returned ballots of which ten favored deletion, three were marked "not voting" and four voted to retain the method. The revision was accordingly submitted to letter ballot of the entire committee and the results of this vote will be reported at the Annual Meeting. It is recommended that this modification be approved for reference to letter ballot of the Society for immediate adoption as standard, and the committee asks for a nine-tenths affirmative vote at the Annual Meeting.

V. REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee will recommend to the Administrative Committee on Standards that the Standard Method of Test for Adhesion of Vulcanized Rubber to Metal (D 429 – 39)[6] be revised and reverted to tentative. The revision consists chiefly of the addition of a stripping method for the evaluation of adhesion of rubber to a single metal plate. This new method has had many applications on war products and is considered a valuable addition to the standard. In addition, a section on adhesion terminology has been added which gives a number of much needed definitions. Subcommittee XX which is responsible for these methods was unanimous in recommending the proposed changes.

VI. TENTATIVES CONTINUED AS TENTATIVE WITHOUT REVISION

The committee recommends that all tentatives under its jurisdiction other than those referred to herein be continued as tentative. Work is under way looking toward revision of a number of these but action must be deferred until this work can be completed.

The recommendations appearing in this report have been submitted to letter ballot[7] of the committee, the results of which will be reported at the Annual Meeting.

ACTIVITIES OF SUBCOMMITTEES

There have been several changes of subcommittee chairmen during the year. F. C. Thorn became chairman of Subcommittee VI on Packings upon the retirement of William Staniar from his active business connection. R. M. Howlett replaced J. A. Baty as chairman of Subcommittee XIX on Immersion Tests. S. H. Tinsley became chairman of Subcommittee XXIV on Tests for Coated Fabrics upon the resignation of A. F. Schildhauer. R. S. Havenhill was appointed chairman of Subcommittee XXV on Low-Temperature Tests following the resignation of B. S. Garvey because of a change in business connection. The loyal service of the retiring subcommittee chairmen and the willing cooperation of the new appointees is greatly appreciated by the committee. All of the subcommittees have been very active during the year and only brief mention can be made here of a few of these activities.

Technical Committee A on Automotive Rubber (J. L. McCloud, chairman, G. H. Swart, secretary) has met at bimonthly intervals during the year and in addition, many of its sections have met separately. Meetings have been well attended and many interesting projects are being carried out. A Proposed Tentative Method of Test for Staining of Organic Finishes by Vulcanized Rubber, referred to earlier in this report, has been developed. Work is nearing completion on another method of test which involves the measurement of the resistivity of electrical conductive rubber. The section on vibration insulators has been very active and is conducting a round-robin testing program on several methods for evaluating the dynamic properties of motor mountings and other vibration insulators. Another round-robin test is in progress on the characteristics of rubber bumpers. A great deal of work has been done on possible revisions of the Tentative Specifications for Rubber and Synthetic Rubber Compounds (D 735). These included the development of tables for plastic materials used in place of rubber in which the committee had the cooperation of Committee D-20 on Plastics. When submitted to letter ballot, the proposed tables were not approved and have been referred back to the section in charge for further consideration. Other extensive revisions of the existing tables are being studied with a view toward simplifying the application of the specifications but this project will probably require considerable time for completion. Specifications for Automotive Coolant Hose which were prepared for the Society of Automotive Engineers have undergone extensive revision during the year. Also, the committee has continued its work on the development of a standard method of testing V-belts and on the preparation of specifications for automobile gaskets and oil seals.

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[6] See Editorial Note, p. 386.

[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Subcommittee IV on Rubber Protective Equipment (Gordon Thompson, chairman) has been reorganized and plans to cover a somewhat wider scope than formerly as is indicated by the change in the title of the subcommittee. The membership of the committee is being extended to make it more representative of all of the interests involved. The personnel of this subcommittee will constitute a Sectional Committee on Rubber Protective Equipment for Electrical Workers, functioning under the procedure of the American Standards Association, with the A.S.T.M. as joint sponsor with the Edison Electric Institute. It will also function as Subcommittee IV of Committee D-11. It is believed that this arrangement will aid greatly in coordinating the standardization work in this very important field.

Subcommittee V on Insulated Wire and Cable (*R. A. Schatzel, chairman*) has had several requests for the development of specifications for small insulated conductors of less than No. 18 A.w.g. with wall thickness of less than $\frac{1}{32}$ in. The advisability of developing specifications for such appliance wire is being considered. The subcommittee also plans to prepare a specification for polyethylene. In addition, the committee expects to resume work on tests for moisture-resistant insulation which was started prior to the war and had to be abandoned because of the pressure of other activities.

Subcommittee XIV on Abrasion Tests (R. H. Taylor, chairman).—In addition to the recommendations in this report, this subcommittee is actively at work investigating tearing tests. Round-robin tests are in progress on different shaped specimens and other details of procedure are being investigated. An extensive study of the literature on the subject has been made. It is anticipated that this work may lead to revisions in the present Standard Method of Test for Tear Resistance of Vulcanized Rubber (D 624 - 44).

Subcommittee XV on Life Tests (H. E. Outcault, chairman) is organizing a new section on ozone aging test procedures in addition to the four sections which were reported last year. This new section was specifically requested by the automotive industry through Technical Committee A who advised that there is an urgent need for a standard testing procedure for evaluating the effects of ozone on rubber.

Subcommittee XIX on Immersion Tests (R. M. Howlett, chairman) has in progress a research project in which the action of C-6 hydrocarbons on rubber is being investigated. A preliminary report has already been made. Consideration is also being given to possible revision of Tentative Methods D 471 including changes which will define more completely the present reference fuels.

Subcommittee XXI on Liquid Rubber Products (J. F. Anderson, chairman) is giving consideration to the addition of the Ford cup method for determination of the viscosity of cements in the present Standard Methods for Viscosity Tests (D 553). Work is being continued also on correlation and reproducibility studies of the Tentative Methods for Testing Rubber Adhesives (D 816).

Subcommittee XXIV on Coated Fabrics (S. H. Tinsley, chairman) has in progress a round-robin test on the suitability of the Du Pont scrub testing machine for evaluating the scrubbing resistance of coated fabrics. Attention is also being given to adhesion tests and to methods for determining abrasion resistance.

Subcommittee XXV on Low-Temperature Tests (R. S. Havenhill, chairman) has started work on investigation of compression set tests at low temperature. It is also actively at work on dynamic tests at low temperatures. A questionnaire has been circulated on dynamic tests and the decision reached to concentrate on the Yerzley oscillograph for a free vibration test but further consideration will be necessary before recommendations can be made on a forced vibration test.

Subcommittee XXVII on Resilience Tests (E. G. Kimmich, chairman) has under consideration a draft of a proposed method for the use of the Yerzley oscillograph in measuring resilience and related properties in compression and shear. Work is being started also on rebound tests of the impact pendulum type and on forced vibration testing. All of this work is of great importance in many applications of rubber for the absorption of vibration but there has been little standardization as yet in this comparatively new field and progress is necessarily rather slow.

This report has been submitted to letter ballot of the committee, which consists of 181 members; 130 members returned their ballots, of whom 118 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

SIMON COLLIER,
Chairman.

ARTHUR W. CARPENTER,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards the recommendation that the Standard Method of Test for Adhesion of Vulcanized Rubber to Metal (D 429 – 39) be revised and reverted to tentative. This recommendation was accepted by the Standards Committee on November 6, 1947, and the new tentative appears in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B, bearing the designation D 429 – 47 T.

APPENDIX

REPORT ON EFFECT OF PLYING-UP COMPRESSION SET SPECIMENS WHEN MAKING COMPRESSION SET TESTS

Report of Task Section of Subcommittee XVII, Consisting of Messrs.

A. E. Juve, F. C. Thorn, A. J. Kearfott, J. F. McWhorter, and L. V. Cooper

According to Tentative Methods of Test for Compression Set of Vulcanized Rubber (D 395 – 46 T),[1] Section 5, the standard test specimen shall be a cylindrical disk ½ in. in thickness. Many commercial products are so thin or so irregularly shaped that a standard specimen cannot be obtained. This necessitates either plying-up portions of the actual product or running the tests on a certified ½-in. slab submitted by the vendor. Many consumers do not like to accept the certified slab and there is

TABLE I.—COMPOSITION OF COMPOUNDS TESTED.
(Parts by weight)

Composition	TM19	TM20	TM21	TM28	TM23	TM24	TM25	TM26
Smoked sheet	100	100						
GR-S					100	100		
GR-M			100	100				
Hycar OR-15							100	100
Zinc oxide	5	5	10	5	5	5	5	5
Sulfur	3	3			2	1.5	1.25	1.5
Stearic acid	1	1	1	0.5			0.5	
Petroleum jelly				2				
FT Black (P-33)								100
SRF Black (Furnex)	65	55	35				65	
EPC Black						60		
HMF Black (Philblack A)					55			
MT Black (Thermax)				106				
Neozone A	1	1	2	2				
Neozone D					1	1		
Brown sub				55				
Suprex clay		45		50				
Paraflux		5						
Light process oil				20				
Light Calcium Magnesia				4				
Di-butyl phthalate							15	
Coumarone indene resin							10	
Bardol B								17
Light mineral oil					10	5		
Cumar MH2½						5		
Monex	0.3				0.5			
Captax						1		
Altax							1.25	
DOTG (Diorthotolylguanidine)							0.25	
DPG (Diphenylguanidine)						0.2		
Santocure		0.75						1
Total	175.3	215.75	152	344.5	173.5	178.7	198.25	224.5

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[1] 1946 Book of A.S.T.M. Standards, Part III-B.

little in the literature concerning the correlation between data obtained on a solid block as compared to a block obtained from plied samples. This report covers the work done by the section appointed to study the effect of plying-up test specimens.

TABLE II.—EFFECT OF PLYING-UP COMPRESSION SET SAMPLE, CURED AT 270 F., AND TESTED AT 158 F.

	Compression Set, per cent			
	Laboratory			
Composition	No. 1	No. 2	No. 3	No. 4
TM-19 low-set rubber				
Solid[a]	13.9	13.15	10.45	14.2
2-ply	14.9	13.5	11.85	14.2
4-ply	17.7	15.75	12.95	17.9
TM-20 high-set rubber				
Solid[a]	29.0	28.0	24.3	30.0
2-ply	30.1	28.25	24.3	32.45
4-ply	32.4	30.5	26.65	31.45
TM-21 low-set neoprene				
Solid[a]	38.2	35.55	31.6	35.6
2-ply	37.5	35.05	33.45	34.2
4-ply	39.2	35.8	35.8	33.15
TM-28 high-set neoprene				
Solid[a]	46.3	46.8	42.25	49.7
2-ply	49.3	46.6	42.3	44.2
4-ply	49.4	47.75	41.65	47.65
TM-23 low-set GR-S				
Solid[a]	10.4	9.35	6.0	8.25
2-ply	11.1	9.4	7.05	8.7
4-ply	13.1	10.75	8.8	10.7
TM-24 high-set GR-S				
Solid[a]	48.6	48.4	44.95	55.7
2-ply	53.3	50.0	46.9	49.7
4-ply	54.4	40.8	48.5	51.7
TM-25 low-set Hycar OR-15				
Solid[a]	26.8	32.4	20.0	26.05
2-ply	22.2	22.1	15.75	21.15
4-ply	20.5	21.5	14.7	21.05
TM-26 high-set Hycar OR-15				
Solid[a]	33.7	32.65	26.65	30.35
2-ply	22.8	26.35	21.8	24.9
4-ply	25.8	25.7	20.45	26.8

[a] Solid = $\frac{1}{2}$ in. thick,
2-ply = $\frac{1}{4}$ in. thick,
4-ply = $\frac{1}{8}$ in. thickand.

At the first meeting of this section it was agreed that the effect of the state of cure was the first variable that had to be ruled out. The second thing that the committee had to take into consideration was the possibility that various stocks might give different relationships. Another problem was the possibility that results might be affected by techniques in use in various laboratories.

After considerable discussion the following basic program was outlined.

1. Eight stocks were to be tested and all were to be about the same hardness. A relatively low permanent set stock and a relatively high permanent set stock would be compounded using smoked sheet in one pair, GR-S, GR-M, and Hycar OR-15 in the other pairs. The compounds finally decided upon are TM19, TM20, TM21, TM28, TM23, TM24, TM25, and TM26. The recipes for these stocks are shown in Table I.

TABLE III.—EFFECT OF PLYING-UP COMPRESSION SET SAMPLE, CURED AT 270 F., AND TESTED AT 212 F.

	Compression Set, per cent			
	Laboratory			
Composition	No. 1	No. 2	No. 3	No. 4
TM-19 low-set rubber				
Solid[a]	30.1	28.65	23.6	28.8
2-ply	34.1	30.4	26.3	32.1
4-ply	35.5	34.4	27.65	32.95
TM-20 high-set rubber				
Solid[a]	61.8	61.85	54.3	62.8
2-ply	63.9	59.5	56.3	64.2
4-ply	66.2	60.75	53.15	65.25
TM-21 low-set Neoprene				
Solid[a]	70.5	66.25	60.05	68.0
2-ply	69.7	65.5	58.7	67.0
4-ply	70.4	66.75	63.8	65.85
TM-28 high-set Neoprene				
Solid[a]	93.5	86.25	85.65	88.6
2-ply	93.5	87.0	83.7	88.7
4-ply	93.0	86.0	87.0	89.4
TM-23 low-set GR-S				
Solid[a]	14.0	13.25	10.3	13.0
2-ply	16.5	13.75	10.7	15.05
4-ply	17.8	15.8	12.1	14.8
TM-24 high-set GR-S				
Solid[a]	80.7	78.75	78.6	80.95
2-ply	88.5	79.5	79.0	82.25
4-ply	86.3	80.15	76.35	81.5
TM-25 low-set HyCar OR-15				
Solid[a]	76.8	71.25[b]	69.3	73.6
2-ply	78.2	67.5[b]	69.1	72.0
4-ply	77.9	71.0	69.3	73.1
TM-26 high-set HyCar OR-15				
Solid[a]	77.4	75.25	74.65	75.85
2-ply	78.5	77.75[b]	70.85	75.9
4-ply	79.4	73.5	70.15	77.5

[a] Solid = $\frac{1}{2}$ in. thick,
2-ply = $\frac{1}{4}$ in. thick, and
4-ply = $\frac{1}{8}$ in. thick.

[b] Average of two determinations more than 5 per cent apart.

2. To rule out the effect of state of cure it was decided that a special technique in obtaining the plied-up buttons would be followed. To obtain a two-ply button two normal height buttons would be drilled out of the standard test slab—one button would be cut just below center and the other button just above center. From the larger portion of the button cut just below center, the upper half of the test button would be obtained by buffing the cut surface side to exactly $\frac{1}{4}$ in. in total height. From the larger portion of the button cut just above center, the cut surface would be buffed so as to obtain a pellet exactly $\frac{1}{4}$-in. high for the lower part of the test specimen. If buffing were done carefully, the resulting specimen would match the original solid $\frac{1}{2}$-in. specimen exactly in state of cure throughout.

TABLE IV.—SECOND SERIES OF TESTS, CURED AT HIGH TEMPERATURE AND AGED AT 158 F.

	Compression Set, per cent			
	Laboratory			
Composition	No. 1	No. 2	No. 3	No. 4
TM-19 (30 at 290 F.)				
Solid[a]	21.9	12.7	13.9	12.2
2-ply	14.7	14.4	14.75	13.7
4-ply	16.2	14.2	15.2	13.5
TM-20 (60 at 290 F.)				
Solid[a]	30.5	33.3	31.7	....
2-ply	32.2	30.0	34.6	....
4-ply	35.5	31.3	33.5	....
TM-20 (60 at 310 F.)				
Solid[a]	23.3	24.4	23.4	23.7
2-ply	27.4	26.5	30.9	24.5
4-ply	28.7	29.5	27.4	26.8
TM-21 (55 at 310 F.)				
Solid[a]	29.0	25.3	28.8	25.0
2-ply	29.5	28.0	28.8	25.4
4-ply	29.3	28.1	30.3	26.2
TM-28 (55 at 310 F.)				
Solid[a]	48.4	43.0	53.0	48.0
2-ply	53.2	50.3	54.6	47.1
4-ply	51.3	53.2	52.5	47.6
TM-23 (40 at 310 F.)				
Solid[a]	17.3	13.0	14.1	12.4
2-ply	12.9	11.3	13.3	11.7
4-ply	13.7	12.7	12.7	11.3
TM-24 (40 at 310 F.)				
Solid[a]	53.6	56.0—	55.4	52.7
2-ply	56.0	55.5	61.3	53.2
4-ply	58.9	56.0	58.2	52.6
TM-25 (30 at 310 F.)				
Solid[a]	58.2	30.6	33.4	27.0
2-ply	24.9	24.6	26.6	22.1
4-ply	22.7	23.3	21.5	20.1
TM-26 (40 at 310 F.)				
Solid[a]	37.7	39.4	43.3	29.3
2-ply	27.8	26.0	30.0	26.3
4-ply	24.4	23.3	25.0	21.0

[a] Solid = $\frac{1}{2}$ in. thick,
2-ply = $\frac{1}{4}$ in. thick, and
4-ply = $\frac{1}{8}$ in. thick.

TABLE V.—SECOND SERIES OF TESTS, CURED AT HIGH TEMPERATURE AND AGED AT 212 F.

	Compression Set, per cent			
	Laboratory			
Composition	No. 1	No. 2	No. 3	No. 4
TM-19 (30 at 290 F.)				
Solid[a]	35.2	30.4	35.8	32.0
2-ply	35.6	31.1	36.9	32.6
4-ply	35.5	31.9	37.4	33.4
TM-20 (60 at 290 F.)				
Solid[a]	70.5	64.5	72.9	....
2-ply	73.5	64.8	78.5	....
4-ply	74.7	67.7	80.4	....
TM-20 (60 at 310 F.)				
Solid[a]	61.3	56.0	65.4	57.0
2-ply	66.6	58.5	68.2	62.0
4-ply	68.3	59.9	70.9	62.7
TM-21 (55 at 310 F.)				
Solid[a]	59.3	53.0	61.7	57.0
2-ply	58.2	52.1	57.4	56.7
4-ply	59.0	51.5	56.0	56.3
TM-28 (55 at 310 F.)				
Solid[a]	91.3	84.7	94.3	87.5
2-ply	93.6	85.6	93.3	87.7
4-ply	94.3	86.6	92.6	87.4
TM-23 (40 at 310 F.)				
Solid[a]	26.5	22.2	25.0	23.2
2-ply	24.7	21.0	24.3	23.2
4-ply	26.0	20.7	25.0	23.3
TM-24 (40 at 310 F.)				
Solid[a]	88.3	81.7	90.7	82.8
2-ply	87.6	82.8	89.0	83.9
4-ply	88.7	82.5	86.7	84.0
TM-25 (30 at 310 F.)				
Solid[a]	84.3	75.5	88.2	79.3
2-ply	78.1	73.7	84.0	77.9
4-ply	82.3	73.9	82.2	77.2
TM-26 (40 at 310 F.)				
Solid[a]	70.6	65.4	75.7	69.0
2-ply	67.0	59.0	66.7	63.4
4-ply	66.8	57.6	69.8	64.3

[a] Solid = $\frac{1}{2}$ in. thick,
2-ply = $\frac{1}{4}$ in. thick, and
4-ply = $\frac{1}{8}$ in. thick.

In obtaining a four-ply button, two buttons were cut and by properly cutting these buttons and properly buffing we were able to get from one button the top ply, and the ply just below center of our final test specimen, and from the second button we were able to get the ply just above center and the lower ply of our final four-ply test specimen. Again, by controlling the buffing temperature we would have the identical state of cure as the solid test button which of course had been cut from the same cured slab.

TABLE VI.

Stock	Cure at 310 F., min.	Durometer Hardness Number	Deflection, per cent	Compression set, per cent	
				Lab. No. 1	Lab. No. 2
TM25					
1. Normal—solid—No buff	30	53	30	33.8	40.5
2. Buffed—top and bottom	30	53	30	33.8	41.7
3. Buffed—top and bottom lubricated	30	53	30	35.8	43.1
4. 2-ply—no buffing top or bottom	30	53	30	36.5	39.0
5. 2-ply—insides not buffed	30	53	30	36.5	39.6
6. 2-ply—all four surfaces buffed	30	53	30	36.5	40.9
7. 4-ply—no buffing—top or bottom	30	53	30	36.5	40.2
8. 4-ply—all surfaces buffed	30	53	30	37.2	41.5
TM26					
1. Normal—solid—no buff	40	60	30	47.4	49.5
2. Buffed—top and bottom	40	60	30	48.0	50.0
3. Buffed—top and bottom lubricated	40	60	30	48.0	49.9
4. 2-ply—no buffing—top or bottom	40	60	30	47.4	48.9
5. 2-ply—insides not buffed	40	60	30	47.4	47.7
6. 2-ply—all four surfaces buffed	40	60	30	48.0	48.4
7. 4-ply—no buffing—top or bottom	40	60	30	50.8	49.4
8. 4-ply—all surfaces buffed	40	60	30	52.0	48.7

3. To insure getting the minimum amount of variation due to varying technique in different laboratories, the following system was followed.

(*a*) All stock was milled in one laboratory.

(*b*) All $\frac{1}{2}$-in. slabs were cured in one laboratory using the optimum cure at two different temperatures: (*1*) 270 F. and (*2*) 290 or 310 F. This optimum cure was arrived at by running a series of cures and selecting the lowest cure giving maximum properties.

(*c*) All test specimens were prepared by one laboratory—the B. F. Goodrich Co.—and this was by far the greatest contribution to the work.

(*d*) The original heights of each test specimen were determined in one laboratory and proper spacer bars were carefully made and sent to each laboratory making the actual permanent set tests.

(*e*) A complete set of test buttons in duplicate was sent to each of four testing laboratories, two of which were producers and two consumers.

(*f*) Permanent set determinations were made using method (*b*) at both 158 and 212 F. at each laboratory. The resulting data are given in Tables II, III, IV, and V.

A careful study of the data in these tables indicates that plying-up the samples gives slightly higher permanent set figures in just over half the tests, but in the remaining tests no difference or the opposite of the above statement is indicated. The exceptions are compounds TM25, and TM26, which when tested at 158 F. indicate that plying-up the sample gives considerably lower permanent set figures. The variation between laboratories even under controlled conditions was quite astonishing.

Explanation of the condition encountered in the Hycar-OR stocks, TM25 and TM26 was not clear and it was decided to recheck carefully the tests on this stock including in the determinations a series of tests comparing buffed and unbuffed portions in contact with the metal set clamp.

Two laboratories cooperated on this test and their findings are shown in Table VI. These figures agree more closely with the findings in the original test on the other stocks and as the figures in Table VI are the average of at least two series of tests, it is felt that the original figures on TM25 and TM26 were in error and should be discarded.

As the result of this work, it was decided to revise Method D 395 – 46 T to allow the use of plied-up samples as recommended in the report of the committee.

REPORT OF COMMITTEE D-12*

ON

SOAPS AND OTHER DETERGENTS

The annual meeting of Committee D-12 on Soaps and Other Detergents was held outside of New York City for the first time in its history in order to participate in the Spring Meeting of the Society in Philadelphia, Pa. Well-attended meetings were held on February 24 and 25, 1947.

There have been a number of changes in membership and representatives. During the past year, eleven new members were added, resulting in a total of 91 members, of whom 42 are classified as producers, 22 as consumers, and 27 as general interest members.

The Advisory Committee voted to reorganize the subcommittee structure to facilitate filing, to make correspondence less cumbersome, and to clarify the functions of the subcommittees. The subcommittees have been divided into three groups, as follows:

Subcommittees on Testing:

T-1	Soaps
T-2	Synthetic Detergents
T-3	Dry Cleaning
T-4	Special Detergents
T-5	Physical Testing
T-6	Metal Cleaning

Subcommittees on Specifications:

S-1	Soap
S-3	Dry Cleaning
S-4	Special Detergents

General Subcommittees:

G-1	Advisory
G-2	Nomenclature and Definitions
G-3	Papers and Publications
G-4	Membership

The above reorganization will become effective after the 1947 Annual Meeting.

Recommendations Affecting Standards

The committee is recommending for publication as tentative a new method for total immersion corrosion test of water-soluble aluminum cleaners and two new specifications for borax and for sodium bicarbonate, withdrawal of tentative revisions of six standard specifications for soap, and the revision of the tentative definitions D 459. The details of these recommendations are covered under Activities of Subcommittees. These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

Activities of Subcommittees

Subcommittees I and II on Methods of Testing and Specifications (J. B. Crowe and F. W. Smither, chairmen). —In Section A of Subcommittee I on Soap Methods it was pointed out that the new edition of the Methods of Soap Analysis of the American Oil Chemists Society contained a few minor changes, most of which are editorial. However, since the Governing Board of AOCS has agreed to form a joint committee with Committee D-12 on soap analysis, it was decided to make no recommendations at this time for changes in the A.S.T.M. methods of soap analysis but to refer the matter to the chairman of the new joint committee. Any new business was also held in abeyance until the joint committee starts to function.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Section A of Subcommittee II on Soap Specifications recommends the withdrawal of the tentative revisions (the former emergency alternate provisions) for Standard Specifications for Chip Soap (D 496 – 39), Olive Oil Chip Soap (D 630 – 42), Powdered Soap (Non-alkaline Soap Powder)(D 498 – 39), Olive Oil Solid Soap (D 592 – 42), Milled Toilet Soap (D 455 – 39), and White Floating Toilet Soap (D 499 – 39), except for the tentative revisions in Section 3 of Specifications D 455 and D 499 changing the requirements for matter insoluble in water, which are to be retained. The tentative revision for Section 3 of Standard Specifications for Compound Chip Soap (with Rosin) (D 690 – 44) changing the basis of calculation from 12 per cent to 15 per cent moisture is also to be retained. Action will be taken at the next meeting on the three tentative revisions being retained.

It was suggested that this section write specifications for "low titer" soap since such a soap is now widely used.

Section C of Subcommittee I and Section D of Subcommittee II on Dry Cleaning Detergents (G. P. Fulton, chairman) discussed proposed specifications for liquid dry cleaning detergents for petroleum solvents. It was agreed that such dry cleaning detergents should be classified into two types depending on total fatty acid content and further classified into three types depending on moisture content, and that the minimum flash point of such a detergent should be 85 F. The fixing of limits on materials other than water was deemed inadvisable; instead, a performance test was proposed based on the ability of the product to emulsify moisture in a petroleum solvent. A method based on this principle will be submitted at the next meeting.

A method of test for fatty acid is being developed.

A representative of a manufacturer of dry cleaning soaps volunteered to make up soaps of known compositions containing the various types of synthetic detergents blended with fatty acids as well as various blends of alcohols and water. (It is believed that alcohol may interfere with the xylol distillation test for water.) These will be tested in several laboratories working in cooperation to determine the effectiveness of the new technique for determining total fatty acids in the presence of synthetic detergents. The samples will also be submitted to moisture determinations including the xylol distillation method, two variations of the acetyl chloride-pyridine method, and two techniques involving the Karl Fischer reagent.

Section D of Subcommittee I on Special Detergents (W. H. Koch, chairman) is planning to provide methods for analysis of borax and of sodium bicarbonate.

Section F of Subcommitee II on Special Detergents (C. C. Zeigler, chairman) is recommending as tentative two new Specifications for Sodium Bicarbonate ($NaHCO_3$)[1] and for Borax (sodium tetraborate, $Na_2B_4O_7 \cdot 10H_2O$).[2]

Section E of Subcommittee I on Physical Testing (J. A. Woodhead, chairman) is investigating physical test methods on lathering properties, pH, wetting, surface and interfacial tension, soiled slide test for dishwashing detergents, and detergency in general. This section is planning to prepare an electrometric pH method based on the Tentative Method for the Determination of the pH of Aqueous Solutions with the Glass Electrode (E 70 – 46 T) A new approach to detergency problems was presented by O. C. Bacon.

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[2] These specifications and methods were accepted as tentative by tne Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

Section G of Subcommittee II on Metal Cleaning (J. C. Harris, chairman) is recommending as tentative a new Method of Immersion Corrosion Test of Water-Soluble Aluminum Cleaners.[2]

This section is investigating indirect performance methods of test for metal cleaners. An instructive lecture on the mechanism of soil removal in metal cleaning was presented at the last meeting of this section by Mr. Samuel Spring, of Frankford Arsenal, Philadelphia, Pa.

Subcommittee III on Nomenclature and Definitions (C. A. Marlies, chairman) recommends that the Tentative definitions of Terms Relating to Soaps and Other Detergents (D 459 - 46 T) be revised as follows:

Current Definitions.—As an editorial change, list the following definitions under the noun, as follows, instead of under the present listing of the adjective:

Detergent, Anionic
Detergent, Cationic
Detergent, Nonionic
Soap, Blended
Soap, Built
Soap, Straight

Add cross-references in alphabetical listing as follows:

Detergent, Synthetic.—See Synthetic Detergent.

Soap.—See first definition.

Change the definitions of "penetrating agent," "washing," and "wetting agent," to read as follows:

Penetrating Agent.—A material which increases the penetration of a liquid medium into a porous material.

Washing.—Cleaning in an aqueous medium.

Wetting Agent.—A material which increases the spreading of a liquid medium on a surface.

New Definition.—Add the following as a new definition:

Foaming Agent.—A material which increases the stability of a suspension of gas bubbles in a liquid medium.

Subcommittee IV on Papers and Publications (W.H. Koch, chairman) reports the publication of a paper on "Evaluation of Surface-Active Agents"[3] by J. C. Harris; also the preparation of a paper by the same author, bringing up to date the Bibliography on Metal Cleaners, which is to be included in the 1947 edition of the Compilation of A.S.T.M. Standards on Soaps and Detergents.

The National Association of Purchasing Agents, under the editorship of W. G. Morse, published the monograph, "Cleaning and Cleaning Materials."[4] This monograph was largely the result of work done by Mr. Morse and members of Committee D-12.

Consumer Specifications.—An extensive discussion at the Philadelphia meeting of consumer specifications for soap products resulted in the decision to have Chairman Van Zile appoint a study group to consider fully the entire subject and to submit a report at the next meeting.

This report has been submitted to letter ballot of the committee, which consists of 92 members; 77 members returned their ballots, of whom 76 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

B. S. VAN ZILE,
Chairman.

J. C. HARRIS,
Secretary.

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[3] This paper was published in two parts: Part I, ASTM BULLETIN, No. 140, May, 1946, p. 76; Part II, ASTM BULLETIN, No. 141, August, 1946, p. 49.

[4] Copies of this monograph may be obtained from the Editor, W. G. Morse, Purchasing Agent, Harvard University, Cambridge, Mass.

REPORT OF COMMITTEE D-13*

ON

TEXTILE MATERIALS

Committee D-13 on Textile Materials returned to its customary prewar practice of holding two meetings during the year. Both were held in New York, N. Y.; the fall meeting on October 16 to 18, 1946, with a registered attendance of 229 members and guests; the spring meeting on March 12 to 14, 1947, with a total attendance of 234. Of the 26 subcommittees and sections of Committee D-13 which deal with standards, 17 held sessions at the fall meeting and 21 at the spring meeting. The setting of general policies and the guidance of the affairs of the committee required two meetings of both the Advisory Committee and the Officers Committee.

The feature of the Papers Session at the fall meeting was a Symposium on Textile Testing in Germany, to which the contributors were Herbert F. Schiefer, Lyman Fourt and Richard T. Kropf.[1] This was the first presentation of information on test methods and machines gathered by the authors while serving in Germany as Scientific Consultants to the Technical Industrial Intelligence Committee of the Foreign Economic Administration.

The Papers Session at the spring meeting was devoted to the use and features of strain gages, and comprised the following three papers: "A Stress-Strain Tester for Textiles, Employing a Magnetic Strain Gage," by J. W. Ballou;[2] "The Application of Resistance Strain Gages to Testing Instruments," by W. M. Murray; and "Measurement and Elimination of Inertia Effects in Textile Testing Equipment by Means of Electric Strain Gages," by H. F. Schiefer.[2]

As a result of further study of the recommendations of the Reorganization Committee, R. H. Brown, chairman, the Advisory Committee has approved the following changes in the organization of Committee D-13: Subcommittee A-1, Section IV on Tire Fabrics becomes a separate Subcommittee A-9 on Tire Cord and Fabrics; the scope of Subcommittee A-2, formerly limited to Rayon and Its Products, has been broadened to include other synthetic fibers and their products, and with appropriate changes in scopes of the sections thereunder; Subcommittee A-3, Section on II on Felt and Subcommittee A-3, Section IV on Pile Floor Coverings have been set up as separate Subcommittees A-10 and A-11, respectively. The Advisory Committee has also authorized the formation of two new subcommittees, one on knit goods, and one on hosiery. The establishment of a subcommittee to deal with bonded fabrics is under consideration.

The places of chairmen who have resigned during the year have been filled as follows: B. L. Whittier as chairman of Subcommittee A-1 on Cotton, vice, K. B. Cook; G. K. Lake as chairman of Subcommittee A-1, Section III on Cotton Woven Fabrics, vice, R. T. Fisher; S. L. Peebles as chairman of Subcommittee A-11 on Pile Floor Coverings, vice, G. Y. Jean; Laura E. Pratt as chairman of Subcommittee A-6 on Household and Garment Fabrics, vice, Ruth O'Brien.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Herbert F. Schiefer, Lyman Fourt and Richard T. Kropf, "Textile Testing in Germany," ASTM BULLETIN, No. 144, January, 1947, p. 17.

[2] "A.S.T.M. Standards on Textile Materials," October, 1947; issued as a separate publication.

C. B. Finckel, one of the charter members of Committee D-13, retired from active business during the year. In view of the 31 years of active and useful service he has rendered in the interest of the committee, he was elected an honorary member.

The members of Committee D-13 wish to record their deep sense of great loss through the untimely death of Dr. Harold DeWitt Smith. Each one who knew him experienced the same profound emotion which always comes with the news of the passing of a close, personal, and beloved friend, for such Dr. Smith had become to all who came into contact with him. He will be remembered, also, as one of those forward-looking scientists in the field of textile research, which the industry can ill afford to lose. His Marburg Lecture on "Textile Fibers, An Engineering Approach to Their Properties and Utilization"[3] delivered before the Society in June, 1944, remains as a monument to his progressive thinking and a challenge to Committee D-13 to carry on in the direction he has pointed.

RECOMMENDATIONS AFFECTING STANDARDS

Committee D-13 is recommending the revision of two tentatives, adoption as standard of tentative revisions of three standards, revision for immediate adoption of three standards, adoption as standard of one tentative and one tentative revision of standard.

The revisions being presented at this time have been submitted to letter ballot,[4] the results of which will be reported at the Annual Meeting. They are referred to under subcommittee activities along with certain other recommendations which will be submitted at a later date to the Administrative Committee on Standards.

All tentatives not specifically referred to are being actively studied by the respective sponsoring subcommittees.

ACTIVITIES OF SUBCOMMITTEES

All subcommittees have been active but only those submitting recommendations will be referred to. Subcommittees are designated in accordance with the recently adopted reorganization of Committee D-13.

Subcommittee A-1 on Cotton and Its Products (B. L. Whittier, chairman):

Section I on Cotton (M. E. Campbell, chairman) recommends an extensive revision of the Tentative General Methods of Testing Cotton Fibers (D 414 - 40 T).[5]

Section II on Cotton Yarns and Threads (W. R. Marsden, chairman) recommends a revision of Standard General Methods of Testing and Tolerances for Cotton Yarns (D 180 - 44)[5] and its reversion to tentative.

Subcommittee A-2 on Rayon and Other Synthetic Fibers and Their Products (A. M. Tenney, chairman):

Section I on Filament and Yarn (J. B. Goldberg, chairman) recommends several revisions in Tentative Methods D 258 - 46 T as given in the Appendix.

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 543 (1944); the Marburg Lecture issued as a separate reprint.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

[5] See Editorial Note, p. 398.

Subcommittee A-3 on Wool and Its Products (A. G. Ashcroft, chairman):

Section I on Wool (Werner von Bergen, chairman) is recommending, for immediate adoption as standard, revisions in the Test for Wool, Hard Scoured in Wool in the Grease (D 584 - 43) as given in the Appendix.

This section is also recommending that the Standard Methods of Test for Fineness of Wool (D 419 - 44)[5] and Standard Specifications and Methods of Test for Fineness of Wool Tops (D 472 - 41)[5] be revised and reverted to tentative. These revised methods include the short fiber method of measuring fineness, which was issued as a tentative revision in June, 1946.

Subcommittee A-8 on Glass Fiber and Its Products (F. S. Mapes, chairman):

This subcommittee recommends a revision of the twist tolerances in Tentative Methods of Testing and Tolerances for Glass Yarn (D 578 - 46 T) as given in the Appendix.

The subcommittee also recommends for immediate adoption as standard, revisions in the breaking strength method in the Standard Methods of Test and Tolerances for Woven Glass Fabrics (D 579 - 44) as given in the Appendix. Also, revisions of the Standard Methods of Test and Tolerances for Woven Glass Tapes (D 580 - 44) to bring the permissible variations of width of glass tapes within practical manufacturing limits and to improve the breaking strength method, as given in the Appendix.

Subcommittee A-10 on Felt (G. H. Harnden, chairman):

This subcommittee recommends for publication new Tentative Specifications for Mechanical Roll Felt.[5]

The subcommittee recommends that the present tentative revisions, except New Section 2, of Standard Methods D 461, be adopted as standard, and that this standard be further tentatively revised to provide for a bursting strength test, as given in the Appendix.

Subcommittee B-1 on Methods and Machines (W. D. Appel, chairman):

Section II on Instruments and Machines (H. J. Ball, chairman) recommends for adoption as standard the tentative revisions of Standard Specifications D 76 - 46.

Subcommittee B-2 on Nomenclature and Definitions (A. G. Scroggie, chairman):

This subcommittee recommends the adoption as standard of several tentative definitions in Part A and the entire Part B of Tentative Definitions D 123 - 46 T.

The subcommittee recommends a revision of Tentative Definitions D 123 - 46 T which adds several new definitions.[5]

The recommendation is also made for the adoption as standard of Tentative Recommended Practice for a Universal System of Yarn Numbering (D 861 - 46 T).

This report has been submitted to letter ballot of the committee, which consists of 330 members, 155 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. J. BALL,
Chairman.

W. H. WHITCOMB,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-13 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

- Mechanical Roll Felt (D 944 - 47 T).

Revision of Tentative:

- Definitions of Terms Relating to Textile Materials (D 123 - 46 T),
- General Methods of Testing Cotton Fibers (D 414 - 40 T), and
- Method of Test for Colorfastness of Dyed Cellulose Acetate Rayon to Atmospheric Fumes (D 682 - 42 T).

Revision and Reversion to Tentative of:

- General Methods of Testing and Tolerances for Cotton Yarns (D 180 - 44),
- Method of Test for Determination of Small Amounts of Copper, Manganese, and Nickel in Textiles (D 377 - 37),
- Methods of Test for Fineness of Wool (D 419 - 44), and
- Spec. and Methods of Test for Fineness of Wool Tops (D 472 - 41).

These recommendations were accepted by the Standards Committee on October 31, 1947, and the new and revised tentatives appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentatives covering textile materials, which are referred to earlier in this report.[1] These standards and tentatives appear in their present form in the 1946 Book of A.S.T.M. Standards, Part III-A.

REVISIONS OF TENTATIVES

Tentative Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 – 46 T):

New Footnote.—Add the following as a footnote reference to the heading "Definitions" of Section 2:

[3] Definitions of twist and other terms used in these methods can be found in the Standard Definitions of Terms Relating to Textile Materials (A.S.T.M. Designation: D 123).

Section 2.—Delete Paragraphs (*d*), (*e*), (*f*), (*g*), (*i*), (*j*), (*k*), and (*l*). Reletter Paragraph (*h*) as (*d*) and add the following new paragraphs:

(*e*) *Standard Atmosphere.*—Air maintained at a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of plus or minus 2 per cent is permitted in relative humidity and plus or minus 2 F. (1.1 C.) in temperature.

(*f*) *Standard Yarn Condition.*—Standard condition shall be that reached by the yarn when it is in moisture equilibrium with a standard atmosphere. Moisture equilibrium must be reached by absorption from a lower regain.

(*g*) *Moisture Equilibrium for Testing.*—The yarn is considered to be in moisture equilibrium suitable for testing when after free exposure to moving air under standard conditions for a period of 1 hr. the increase in weight of the sample does not exceed 0.1 per cent.

(*h*) *Commercial Regain.*—The commercial moisture regain shall be 11 per cent for regenerated cellulose rayon yarns and 6.5 per cent for cellulose acetate rayon yarns.

Reletter Paragraphs (*m*) and (*n*) as (*i*) and (*j*), respectively.

Sections 7 to 11, inclusive.—Delete these sections and add the following as a new Section 7:

7. Methods for the identification of the common types of rayon are given in Tentative Methods for Identification of Fibers in Textiles (A.S.T.M. Designation: D 276).

Sections 12 to 21, inclusive.—Renumber as Sections 8 to 17, respectively.

Present Section 19(*b*).—In line 13, change "18 in." to read "18 or 10 in.," and in line 21 change "20 in." to read "20 or 10 in."

Sections 22 to 26, inclusive.—Delete these sections and renumber Sections 27 to 37 as Sections 18 to 28, respectively.

Tentative Methods of Testing and Tolerances for Glass Yarn (D 578 – 46 T):

Section 5 (*b*).—Change to read as follows and delete the appended note:

(*b*) *Turns.*—The average twist of the yarn shall not vary beyond the following limits:

Turns per in.	Permissible Variations, plus or minus
Under 1	0.5 turns per in.
1 to 7.5 incl.,	20.0 per cent
Over 7.5 to 10, incl.,	10.0 per cent
Over 10	5.0 per cent

TENTATIVE REVISION OF STANDARD

Standard Methods for Testing Felt (D 461 – 45):

New Section.—Add the following as a

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[1] See p. 395.

new Section 10 to follow the section on breaking strength:

10. The bursting strength test shall be made with an approved type of diaphragm bursting tester in accordance with the procedure prescribed in Section 11 of Standard Methods of Test for Knit Goods (A.S.T.M. Designation: D 231).

REVISION OF STANDARDS, IMMEDIATE ADOPTION

Standard Methods of Test for Hard Scoured Wool in Wool in the Grease (D 584 - 43):

Section 1.—Change to read as follows:

1. This method of test covers the procedures for determining the percentage of hard scoured wool content of individual fleeces or similar quantities of grease wool on a laboratory scale. It is applicable to both apparel wool and carpet wool.

Section 2.—Delete Paragraphs (*b*) and (*c*) and change Paragraph (*a*) to read as follows:

2. *Hard Scoured Wool.*—Hard scoured wool shall be scoured wool containing 12 per cent of moisture (equivalent to 13.64 per cent moisture regain), an alcohol extractable matter content of 1.5 per cent, and an ash content of 0.5 per cent.

Section 5.—Delete this section.

Section 6.—Renumber as Section 5 and in Paragraph (*c*), last sentence delete "ether extractable matter" and renumber "Sections 7, 8, and 9" as "Sections 6 and 7."

Section 7.—Renumber as Section 6 and change title from "Ether Extraction" to read "Alcohol Extraction." In the first sentence, change "alcohol free ethyl ether" to read "neutral 95 per cent alcohol (Note)." In the second sentence, substitute "neutral alcohol" for "ethyl ether." Change the last sentence and Note to read as follows: "The alcohol then shall be evaporated from the extract and the residue dried at 100 to 105 C. to constant weight." "*Note.*—Denatured neutral alcohol may be used for the extraction provided it leaves no residue when 100 ml. of the denatured alcohol is evaporated and dried at 100 to 105 C."

Section 8.—Delete this section.

Section 9.—Renumber as Section 7. Delete the present wording and substitute the revised wording of the text and Note appended to the standard,[2] changing the second sentence of the text to read: "... silica crucible having approximately the following dimensions: ..."

Section 10 (a).—Renumber as Section 8 and substitute "extractives" for "extractions" and delete "(a)" after "Section 2."

Section 10 (b).—Line 2, insert "content" after "wool"; lines 3 and 4, delete "ether extract" and substitute "extractives" for "extract"; line 6, change "Eqs. 1 to 3" to "Eqs. 1 and 2"; line 8, change "Eq. 4" to read "Eq. 3"; line 11, change "Eq. 5" to read "Eq. 4"; line 13, change "Eq. 6" to read "Eq. 5"; line 15, change "Eq. 7" to read "Eq. 6"; substitute the following equations for the present equations 3, 4, 5, 6, and 7:

$$P = \frac{E \times (100 - X - Y) \times 100}{D \times (100 - 12 - 1.5 - 0.5)} \quad \ldots (3)$$

$$Q = \frac{P \times C}{100} \quad \ldots\ldots\ldots\ldots (4)$$

$$R = \frac{E_t \times (100 - X_t - Y_t)}{100 - 12 - 1.5 - 0.5} \quad \ldots\ldots (5)$$

$$S = \frac{(Q + R) \times 100}{F} \quad \ldots\ldots\ldots (6)$$

In the legend make the following changes:

In *A* substitute "alcohol" for "ether"; Delete *B*; Change Sections 6, 7 and 9, to read Sections 5, 6, and 7 respectively wherever they occur; in S insert "of" after "percentage"; change "W_a" to read, "weight of oven-dry scoured wool or tags used for determination of extractable matter in test specimen or tags"; change "W_c" to read "W_b" (also in Eq. 2) and change to read, "weight of oven-dry scoured wool or tags used for determination of ash in test specimen or tags"; in "X, Y, Z" and "X_t, Y_t, Z_t," delete "Z" and "Z_t" and the words "ether" and "alcohol extractable matter" in each case.

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[2] This revision refers to the tentative revision submitted in June, 1946, 1946 Book of A.S.T.M. Standards Part III-A, p. 1261.

Section 11.—Renumber as Section 9. In line 3, change the words "Section 10 differ by" to read "Section 8 have a range of," and in the last line, change "Section 6 (*c*)" to read "Section 5 (*c*)."

Standard Methods of Testing and Tolerances for Woven Glass Fabrics (D 579 - 44):

Section 11 (*b*).—Change to read as follows:

(*b*) *Test Sample.*—Two samples, approximately 8 by 10 in., shall be cut from the fabric, one with the warp yarns and the other with filling yarns parallel to the 8-in. dimension. Each sample shall be laid on a piece of medium weight wrapping paper of similar size. Five test specimens, 1½ × 6 in., shall be laid out on the fabric by drawing light lines with a soft black wax pencil so that the yarns to be tested, warp or filling, shall be parallel to the longer dimension. Lines shall be drawn across the specimens, 1⅝ in. from each end, using very light pressure of the wax pencil to avoid possible damage to the surface filaments. The 1⅝-in. strips shall be thoroughly impregnated with a methacrylate solution (Note 1) which must soak through the fabric in order to secure firm adhesion to the paper (Note 2) and be spread in an even film to secure a uniform pressure from the testing machine jaws against the test specimen. The impregnated sample shall be allowed to dry slowly, 24 hr. without forced drying, until the solvent is completely removed.

NOTE 1.—The methacrylate solution shall be prepared by mixing:

45 per cent by weight of butyl methacrylate,
55 per cent by weight of xylene or toluene, and
a small amount of an oil-soluble red dye.

The viscosity of this solution should be about 3000 centipoises, approximately that of honey at room temperature. It may be necessary to change the consistency for some types of fabrics to permit complete penetration of all interstices and to prevent capillary migration of the solution along the yarns into the test area.

NOTE 2.—It is advisable to have the impregnant cover the cross lines to reinforce these sections where some of the surface fibers may have been fractured when these lines were drawn. A sign writer's brush, ⅝ in. in width with 1-in. white fitch bristles, is very satisfactory for applying the compound. On heavy fabrics it is advisable to paint both sides of the specimens either by applying a coat of the impregnant to the back of fabric or to the top surface of the backing paper.

(*c*) *Test Specimen.*—Cut the 6 by 1½-in. test specimen strips from the prepared sample (Paragraph (*b*)), without removing the paper backing. The central unimpregnated portion of the specimen, 2¾ in. in length shall then be raveled to 1 in. in width if there are more than 30 yarns per in., or to the nearest whole number of yarns per inch if there are 30 or less yarns per inch in the fabric as determined by count. The end sections shall be left 1½ in. wide. After raveling, the 1½-in. wide paper backing shall be cut across midway between the ends.

(*d*) *Breaking.*—The clamps of the testing machine shall be set 3 in. apart and the specimen inserted so that the yarns under test will be parallel to the direction of pull (Note 3). To minimize crushing and cutting of the treated specimen ends by the steel jaws of the clamps, the inside surfaces of the jaws shall be lined with thin cardboard of good quality (0.010 to 0.015 in. in thickness), securely fastened with ½-in. wide strips of scotch tape applied over the ends of the jaws. The test result shall be discarded if a specimen slips in the clamps, breaks in the clamps, breaks at the edge of the clamps, or has a strength which falls considerably below the average for the other specimens, and another specimen shall be substituted. In the case of specimens containing 30 or less yarns per inch, the average breaking strength per yarn shall be calculated and then multiplied by the exact number of yarns per inch as determined by a yarn count.

NOTE 3.—The yarns in a glass fabric have a low inherent elongation at the breaking load. This lack of elasticity necessitates the careful insertion of the specimen in the clamps in order to provide an equal loading on all the yarns. When a specimen is skewed between the clamps, the tension on the yarns will vary across the raveled strip and an erroneous low breaking strength value will result.

Section 11 (*c*).—Reletter as Paragraph (*e*) and change to read as follows:

(*e*) At least five breaking strength determinations shall be made in the direction of the warp and five in the direction of the filling and the average of the five tests shall be the breaking strength of the fabric warpwise and fillingwise, respectively.

Standard Methods of Testing and Tolerances for Woven Glass Tapes (D 580 – 44):

Section 4.—Change to read as follows: "4. The width of the tape shall not vary from that specified by more than plus or minus $\frac{1}{16}$ in."

Section 15 (*b*).—Change last sentence to read as follows: "In order to prevent breakage of the specimen at the clamps, it is recommended that the specimen be impregnated and handled in accordance with the prescribed procedure for determination of breaking strength in Section 11 of Standard Methods of Testing and Tolerances for Woven Glass Fabrics (A.S.T.M. Designation: D 579)."

REPORT OF COMMITTEE D-14*

ON

ADHESIVES

Committee D-14 on Adhesives and its subcommittees held two meetings during the year following a three-day meeting in Buffalo, N. Y., at the 1946 Annual Meeting. At the Buffalo meeting a talk was given by W. A. Weyl on "The Theoretical Basis of Adhesion."[1]

A two-day meeting at Atlantic City, N. J., on October 15 and 16 produced considerable activity in reference to test methods and other technical problems, including a very interesting talk by W. O. Baker entitled, "Cohesion, Adhesion and Structure of Polymer Molecules."

A two-day meeting at Philadelphia, Pa., on April 24 and 25, 1947, completed the meetings of Committee D-14 for the year. At this session a talk was given by A. G. H. Dietz on "Recent Swiss Studies on the Fundamental Properties of the Strength of Adhesives."

At present Committee D-14 consists of 87 members of whom 30 are classified as consumers, 42 as producers, and 15 as general interest members. There are 13 alternates for company and individual members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1946 Annual Meeting, Committee D-14 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods of Test for:

Resistance of Adhesive Bonds to Chemical Reagents (D 896 – 46 T), and

Tensile Properties of Adhesives (D 897 – 46 T).

These recommendations were accepted by the Standards Committee on September 9, 1946, and the tentative methods appear in the 1946 Book of A.S.T.M. Standards, Part III-B.

On December 5, 1946, the Standards Committee accepted the Tentative Methods of Test for Peel or Stripping Strength of Adhesives (D 903 - 46 T). The new tentative appears in the 1946 Book of A.S.T.M. Standards, Part III-B.

The proposed Tentative Recommended Practice for Determining the Effect of Artificial (Carbon-Arc Type) and Natural Light on the Permanence of Adhesives was accepted as tentative by the Standards Committee on December 31, 1946. The new tentative recommended practice was assigned the designation D 904 – 46 T, and appears in the 1946 Book of A.S.T.M. Standards, Part III-B.

On February 24, 1947, the Standards Committee accepted the following recommendations:

Tentative Methods of:

Test for Determining the Applied Weight Per Unit of Dried Adhesive Solids (D 898 – 47 T),

Test for Determining the Applied Weight Per Unit Area of Liquid Adhesives (D 899 – 47 T),

Testing the Strength Properties of Adhesives in Shear by Compression Loading (D 905 – 47 T), and

Testing the Strength Properties of Adhesives in Plywood Type Construction in Shear by Tension Loading (D 906 – 47 T).

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1506 (1946); also available as a separate reprint.

These new tentatives appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B.

The proposed Tentative Definitions of Terms Relating to Adhesives was accepted by the Standards Committee on May 7, 1947. The new tentative appears in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B bearing the designation D 907 – 47 T.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Properties (M. H. Bigelow, chairman) has been very active. Several test methods have already been accepted as tentative by the Standards Committee. At the October meeting M. H. Bigelow returned to the chairmanship relieving A. G. H. Dietz who served very effectively as acting chairman during Mr. Bigelow's period of military service.

The Section on Tensile Strength is studying the possibility of expanding the tension test method to include other materials than wood and metal. The Section on Shear Strength is actively considering test methods which will produce a more nearly pure shear measurement than those now in existence. The Section on Impact Strength has prepared a test method[2] which has been approved by letter ballot of the full committee. A very extensive fatigue study has been made by the Section on Fatigue Strength and this work was reported at the Philadelphia meeting. The Section on Flexural Strength has developed a method for testing laminated assemblies which has received approval of the subcommittee and is destined for letter ballot of the full committee. A Section on Cleavage as differentiated from peel has done some interesting work including round-robin tests. This study is nearing completion. A new Section on Flexibility has been established to develop methods for measuring flexibility characteristics of adhesive films.

Subcommittee II on Analytical Tests (L. Repsher, chairman) has been temporarily inactive pending specific requests for work.

Subcommittee III on Permanence (F. J. Wehmer, chairman) has had two tentative methods accepted by the Standards Committee during the past year.

The Section on Effect of Moisture and Temperature has been actively studying both cyclic and continuous exposure conditions. A series of tests is now in progress. The Section on Effect of Chemical Factors has completed one test method and is now considering other ramifications of the subject. The Section on the Effect of Light has had one method approved and is now studying other test methods. The Section on the Effect of Biological Factors has done extensive amounts of testing and is accumulating data towards preparing a test method.

Subcommittee IV on Working Properties (C. B. Hemming, chairman) has had two test methods accepted by the Standards Committee during the past year. These were developed by the Section on Coating or Spreading Rate. The Section on Tack and Blocking has been very active in a study of methods for determining tack. The Section on Cure has recently been reorganized and is making a serious study relative to cure properties.

Subcommittee V on Specifications (G. Reinsmith, chairman) has outlined methods of classification for adhesives. Several possible formats will be sent out for letter ballot of the full committee. The preparation of the specifications themselves must await the development of further test methods by other subcommittees.

Subcommittee VI on Nomenclature and Definitions (G. M. Kline, chairman) prepared the definitions of 22 terms in the Tentative Definitions of Terms Relating to Adhesives (D 907 – 47 T) accepted by the Standards Committee. A second list of definitions is now ready for letter ballot and a third list is under consideration.

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[2] See Editorial Note, p. 405.

Subcommittee VII on Research (A. G. H. Dietz, chairman) has been recently established to study some fundamental adhesive problems. These are such matters as the rate of load application during testing, preparation of surfaces for adhesives, the nature of adhesive bonds, physical strength properties *versus* geometry of bonds, and determination elastic constants such as modulus of elasticity.

This report has been submitted to letter ballot of the committee which consists of 87 members; 61 members returned their ballots of which 59 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

R. C. PLATOW,
Chairman.

L. P. HART, JR.,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-14 presented to the Society through the Administrative Committee on Standards the proposed Tentative Method of Test for Impact Strength of Adhesives. The new tentative method was accepted by the Standards Committee on October 31, 1947, and appears in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B, under the designation D 950 – 47 T.

REPORT OF COMMITTEE D-16[*]

ON

INDUSTRIAL AROMATIC HYDROCARBONS

Meetings of subcommittees, Advisory and General Membership of Committee D-16 on Industrial Aromatic Hydrocarbons were held June 27, 1946, in Buffalo, N. Y., during the Annual Meeting of the Society. The Advisory Group also met February 27, 1947, at the time of the A.S.T.M. Spring Group Committee Meetings in Philadelphia, Pa.

I. New Tentative

While specifications covering specific sulfur compounds apparently are not highly essential at this time, Committee D-16, nevertheless, recognizes that sulfur compounds play an important part, particularly in catalytic processes, and the use of refined aromatic hydrocarbons in such processes appears certain to involve some process troubles, which will raise questions concerning specific sulfur compounds. In preparation for such eventualities, Section 5E on Sulfur Compounds (A. W. Beshgetoor, chairman) was constituted within Subcommittee II on Methods of Test for Refined Aromatic Products. This section has developed a proposed Tentative Method of Test for Thiophene in Benzene as appended hereto,[1] which the committee recommends for publication as tentative. This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

II. Adoption of Tentatives as Standard

The following tentative specifications and methods prepared by Committee D-16 have stood for two years without change:

Tentative Specifications for:[3]

Nitration Grade Benzene (D 835 - 45 T),
Industrial Grade Benzene (D 836 - 46 T),
Industrial 90 Benzene (D 837 - 45 T),
Refined Solvent Naphtha (D 838 - 45 T),
Crude Light Solvent Naptha (D 839 - 45 T),
Crude Heavy Solvent Naptha (D 840 - 45 T),
Nitration Grade Toluene (D 841 - 45 T),
Industrial Grade Toluene (D 842 - 45 T),
Nitration Grade Xylene (D 843 - 45 T),
Industrial Grade Xylene (D 844 - 45 T),
Five-Degree Xylene (D 845 - 45 T), and
Ten-Degree Xylene (D 846 - 45 T),

Tentative Methods of Test for:[3]

Acidity of Benzene, Toluene, Xylenes, Solvent Napthas, and Similar Industrial Aromatic Hydrocarbons (D 847 - 45 T),
Acid Wash Color of Benzene, Toluene, Xylenes, Refined Solvent Napthas, and Similar Industrial Aromatic Hydrocarbons (D 848 - 45 T),
Copper Corrosion of Industrial Aromatic Hydrocarbons (D 849 - 45 T),
Distillation of Industrial Aromatic Hydrocarbons (D 850 - 45 T),
Paraffins in Industrial Aromatic Hydrocarbons (D 851 - 45 T),
Solidifying Point of Benzene (D 852 - 45 T), and
Color, and Hydrogen Sulfide and Sulfur Dioxide Content (Qualitative) of Industrial Aromatic Hydrocarbons (D 853-45T).

The above specifications and methods are now recommended for approval for reference to letter ballot of the Society for adoption as standard. This recommendation has been submitted to letter ballot of the committee.[2]

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] This method has been accepted as tentative by the Society and appears in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

[3] 1946 Book of A.S.T.M. Standards, Part III-A.

III. ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Methods of Test for Refined Aromatic Products (V. J. Altieri, chairman).—A review has been made by Section 1 on Sampling (F. M. Thatcher, chairman) of existing standard practice in the field of sampling and proposed Tentative Methods for the Sampling of Industrial Aromatic Hydrocarbons have been assembled. These methods are now in established channels preparatory to recommendation for publication as tentative.

Cooperative testing designed to evaluate the usefulness of Tentative Methods D 847 to D 853 has been completed on five aromatic hydrocarbons (1 benzene, 2 toluenes and 2 xylenes). The data assembled are now being analyzed by Section 3C on Cooperative Tests (R. B. Greene, chairman). This subcommittee has consolidated its Sections 2A on Paraffins and 2B on Solidifying Point (J. N. Roche, chairman) with Section 3C on Cooperative Tests inasmuch as these sections have completed their tasks to the point that future activities can be done by the latter group.

A general review is under way by Section 5E on Sulfur Compounds (A. W. Beshgetoor, chairman) to determine the types of sulfur compounds one would expect to find in industrial aromatic hydrocarbons and subsequently to develop methods by which they can be determined. As a start in this endeavor, this section has prepared the proposed Method of Test for Thiophene in Benzene as previously mentioned in this report.

Arrangements have been made for various cross memberships with Committee D-3 on Gaseous Fuels, as there is joint interest in work relating to the determination of special constituents of gaseous fuels since such constituents are also found in industrial aromatic hydrocarbons.

This report has been submitted to letter ballot of the committee which consists of 35 voting members; 21 members returned their ballots, of whom 20 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

D. F. GOULD,
Chairman.

W. L. DOUTHETT,
Secretary.

REPORT OF COMMITTEE D-18*
ON
SOILS FOR ENGINEERING PURPOSES

Committee D-18 on Soils for Engineering Purposes held two meetings during the year, one at Buffalo, N. Y., on June 26, 1946, and one at Cincinnati, Ohio, on March 27, 1947. The Advisory Committee held a meeting in Washington, D. C., on December 4, 1946.

Since the long-established Symposium Committee, under the leadership of its late chairman, F. C. Lang, had accomplished its purpose when the compilation "Procedures for Testing Soils" was published, a newly-created Symposium Committee, with W. S. Housel as chairman, was charged with the preparation of a "Symposium on Load Testing" to be held at the 1947 Annual Meeting. F. J. Converse and Hamilton Gray are serving as the other members of this committee. The symposium consists of the following papers:[1]

"Field Loading Tests for the Evaluation of the Wheel Load Capacities of Airport Pavements" by L. A. Palmer;

"Methods of Testing Soils for Runways and Foundations" by Elwyn E. Seelye, W. D. Bailey and S. D. Teetor;

"The Use of Load Tests in the Design of Flexible Pavements" by W. H. Campen and J. R. Smith;

"A Cyclic Load-Test Procedure" by Jean E. Hittle and W. H. Goetz;

"A Canadian Investigation of Load Testing Applied to Pavement Design" by Norman W. McLeod; and

"Field Bearing Tests Applied to Pavement Design" by Robert R. Philippe.

A Special Papers Committee with M. D. Catton as chairman and E. F. Bennett and C. W. Allen as members has assembled a group of papers of general interest to be presented at the Annual Meeting in June, as follows:

"The Use of the Maximum Principal Stress Ratio as the Failure Criterion in Evaluating Triaxial Shear Tests on Earth Materials,"[2] by W. G. Holtz;

"Uplift Pressure on Bridge Foundations in Clay Revealed by Shear Tests,"[3] by G. O. Kerkhoff and W. S. Housel; and

"Tests for Thermal Diffusivity of Granular Materials,"[4] by William L. Shannon and Winthrop A. Wells.

A special committee consisting of the members of the Advisory Committee of Committee D-18 and Mr. E. F. Bennett, Principal Soils Engineer, New York State Department of Public Works, was appointed, under the chairmanship of K. B. Woods, to consider the reorganization of Committee D-18. Plans have been submitted and have received considerable discussion in meetings of the Advisory Committee. The proposed plan for reorganization of Committee D-18, together with By-laws rewritten in conformity therewith, are being submitted to the main committee.

At the meeting of the Advisory Committee held in Washington, D. C. on December 4, 1946, the resignation of K. F. Vernon as Secretary was presented and accepted. The transfer of Mr.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] To be issued later as a separate publication.

[2] See p. 1067.

[3] See p. 1044.

[4] See p. 1065.

Vernon to a new assignment in the Bureau of Reclamation outside of Washington prevents his further activity in this capacity. Mr. R. F. Blanks was appointed Acting Secretary. At his request Mr. Blanks was relieved of the chairmanship of Subcommittee VIII on Methods of Testing Soil Under Triaxial Loading; and W. G. Holtz, Head of the Earth Materials Laboratory, Bureau of Reclamation, Denver, Colorado, was appointed chairman of this subcommittee to succeed Mr. Blanks.

The committee recommended the immediate reprinting of the compilation "Procedures for Testing Soils" in its present form. It further recommended the publication of a supplement containing later information on soil test procedures as soon as practicable. The general revision of the compilation itself is considered to be an extensive undertaking and a committee will be appointed for this task.

I. NEW TENTATIVES

The committee recommends the following two new methods for publication as tentative:

Tentative Methods of:

Testing Soil-Bituminous Mixtures, recommended jointly with Committee D-4 on Road and Paving Materials,[5] and

Test for Shear Strength of Flexible Road Surfaces, Subgrades, and Fills by the Burggraf Shear Apparatus,[5] as published by the Society in "Procedures for Testing Soils," September, 1944, p. 119; also recommended jointly with Committee D-4.

II. ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Method of Test for Cement Content of Soil-Cement Mixtures (D 806 - 44 T)[6] be approved for reference to letter ballot of the Society for adoption as standard.

III. CONTINUATION OF TENTATIVES WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Specific Gravity of Soils (D 854 - 45 T) and the Tentative Definitions of Terms and Symbols Relating to Soil Mechanics (D 653 - 42 T) be continued as tentative without revision.

The committee also recommends that the Tentative Method of Test for Moisture-Density Relations of Soils (D 698 - 42 T) be continued as tentative as revisions of this method are under consideration by the joint subcommittee of Committees D-4 and D-18.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions recommended the retention of the Tentative Definitions of Terms and Symbols Relating to Soil Mechanics (D 653 - 42 T) as tentative. Chairman Kimball was relieved of the chairmanship of this subcommittee at his request. The appointment of Mr. Kimball's successor is held in abeyance pending the reorganization of the committee.

Subcommittee III on Methods of Sampling Soils (C. W. Allen, chairman) organized two groups to investigate the soil dynamics and aerial photographic methods for making soil profiles. The proposed revisions of the Standard Methods of Surveying and Sampling Soils for Highway Subgrades (D 420 - 45) were submitted to the committee members.

Subcommittee IV on Methods of Testing the Physical Characteristics of Soils (Harold Allen, chairman) has in preparation a revision of Method D 698 and a new method of test for density of soil in place.

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[5] These methods were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part II.

[6] 1946 Book of A.S.T.M. Standards, Part II.

[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M Headquarters.

Subcommittee IV recommends that the Method of Test for Specific Gravity of Soils (D 854 - 45 T) be continued as tentative since less than two-thirds of those voting on recent letter ballot were in favor of its adoption as standard.

The subcommittee is considering means for expediting the review and revision of standard and tentative methods of testing coming under its jurisdiction, by the organization of sections within the subcommittee, each of which would be responsible for the critical review of one or more of these methods.

Subcommittee VIII on Methods of Testing Soil Stability under Triaxial Loading (W. G. Holtz, chairman) completed its reorganization during the past year and now has a limited membership of eight to increase the efficiency of the group. All available data on triaxial load testing which the members had obtained during the past five years are being assembled.

Subcommittee XI on Methods of Testing Drainage Properties of Soils (K. B. Woods, chairman) has made the following recommendations to the Advisory Committee:

1. That a small research subcommittee be appointed to study electroendosmosis, if there are sufficient members of Committee D-18 interested in activity in this field,

2. That a research subcommittee be assigned the task of developing a method of test for measuring the permeability of granular and semigranular materials,

3. That the appropriate subcommittee develop specifications for backfill material for French drains and other roadway drainage, and

4. That an appropriate subcommittee be assigned the task of developing specifications for base courses.

Joint Subcommittee of Committees D-4 and D-18 on Tests and Specifications for Stabilized Soils (H. F. Clemmer, chairman) held meetings on December 4, 1946 and March 26, 1947, both meetings being well attended. The work of this committee had been quite static during the emergency years, but the subcommittee has been able to plan a program and organize sections which will provide data and information of value.

Besides sections dealing with various materials for stabilization, that is, asphalt, tar, asphalt emulsion, cement, and granular material for stabilized mixtures, there are four special sections as follows:

Compaction, Density and Moisture in Soils, Harold Allen, chairman

Permeability and Drainage of Granular Stabilized Soils Mixtures, E. F. Bennett, chairman

Method of Test to Determine the Strength of Granular Stabilized Mixtures, H. H. Miller, chairman, and

Moisture-proofing Ability of Various Admixtures, E. A. Willis, chairman.

Though none of these special sections had reports to present other than progress, they are active.

The status of the various methods of testing and specifications under the supervision of this joint subcommittee were considered. The Joint Subcommittee recommended that the Tentative Method of Test for Cement Content of Soil-Cement Mixtures (D 806 - 44 T) be adopted as standard.

Consideration was given to the adoption as standard of Specifications D 556 - 40 T and D 557 - 40 T, Materials for Stabilized Base Courses, and Materials for Surface Courses, respectively; however, due to various suggestions for changes, it is recommended that these specifications be maintained as tentative.

The Joint Subcommittee recommended as tentative the Method of Test for Shear Strength of Flexible Road Surfaces, Subgrades, and Fills, as mentioned earlier in this report. Also recommended was the publication as tentative of the Method of Testing Soil-Bituminous Mixtures, as mentioned earlier in this report. This method was prepared by a group representing producers of typical bituminous materials used for the stabilization of highway soils. Its development and application were discussed in a paper[8] at the 1946 Annual Meeting.

This report has been submitted to letter ballot of the committee which consists of 80 voting members; 48 members returned their ballots of whom 47 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. J. KILCAWLEY,
Chairman.

R. F. BLANKS,
Acting Secretary.

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[8] E. O. Rhodes, and P. F. Phelan, "Development of Method of Test for Soil-Bituminous Mixtures," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1416 (1946).

REPORT OF COMMITTEE D-19[*]

ON

WATER FOR INDUSTRIAL USES

Committee D-19 on Water for Industrial Uses and its subcommittees held two meetings during the year: one at Buffalo, N. Y., June 26 to 28, 1946, and the other at Philadelphia, Pa., February 27 and 28, 1947.

The committee is sponsoring a Session on Water at the 1947 Annual Meeting of the Society at Atlantic City, N. J., at which the following papers will be presented:

"An Evaluation of Test Methods for the Determination of Dissolved Oxygen in Deaerated Boiler Feed Water,"[1] by J. F. Sebald, Worthington Pump and Machinery Corp., Harrison, N. J.

Introduction[2] by C. E. Imhoff, Allis Chalmers Mfg. Co., Milwaukee, Wis., to the Round-Table Discussion on the Identification of Water-Formed Deposits.

"Use of the Spectroscope in the Determination of Constituents of Boiler Scale and Related Compounds,"[3] by Alton Gabriel, Howard Jaffe, and Maurice Peterson, U. S. Bureau of Mines, Washington, D. C.

"Some Problems in Nomenclature in Mineralogy and Inorganic Chemistry,"[4] by Michael Fleischer, U. S. Geological Survey, Washington, D. C.

The committee has in active preparation the contents of a projected A.S.T.M. Manual on Industrial Waters. The Manual will include chapters on the following subjects:

I. Composition of Industrial Water Supplies and Water-Formed Deposits,
II. Uses of Industrial Water,
III. Difficulties Caused by Water in Industry,
IV. Treatment of Water for Industrial Use,
V. Sampling of Industrial Water and Water-Formed Deposits,
VI. Analysis of Industrial Waters,
VII. Indentification of Water-Formed Deposits,
VIII. Analysis of Water-Formed Deposits, and
IX. Appendix.

Max Hecht, the Society's representative on the Feedwater Subcommittee of the Boiler Code Committee, American Society of Mechanical Engineers, attended a meeting of the working group at which the third draft of Section VII-B on Suggested Care of Stationary Steam Boilers was completed. Members of Committee D-19 were advised of the status of this assignment and instructions were given to the society representative to vote for acceptance of the draft.

A meeting of the Executive Subcommittee of the Joint Research Committee on Boiler Feedwater Studies was held in New York, N. Y., on December 5, 1946. R. E. Hall, secretary of Committee D-19 represented the Society, serving for Max Hecht and F. N. Speller.

I. New Tentative Methods

The committee recommends for publication as tentative the following three methods and one recommended practice:

Tentative Method for Detection of Iron Bacteria in Industrial Waters.[5]—This is the first of a series of methods using the biological microscope for the detection of biological products that form deposits on surfaces in contact with industrial waters. The glossy prints of photomicrographs of iron bacteria which are used in this recommended practice were secured through the courtesy of Dr. R. L. Starkey, Associate Microbiologist, New Jersey Agricultural Experiment Station, New Brunswick, N. J.

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] See p. 1120.

[2] See p. 1087.

[3] See p. 1110.

[4] See p. 1089.

[5] These methods and recommended practices were accepted as tentative by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

Tentative Recommended Practice for Application of X-Ray Diffraction Methods to Water-Formed Deposits.[5]—This method describes the procedure for the identification of compounds that form deposits and scale on surfaces in contact with industrial water.

Tentative Method of Reporting Results of Chemical Analysis of Water-Formed Deposits.[5]—This method furnishes information for the reporting of the results of the chemical analysis of water-formed deposits and includes accepted definitions of various types of such deposits. It will be supplemented at a later date with methods for the reporting of results of inspection using the various types of microscopes and analysis by the spectrograph and X-ray diffraction.

Tentative Recommended Practice for Corrosion Tests in Industrial Waters (NDHA Method).[5]—This recommended practice describes the procedure for observing the corrosivity of industrial waters, particularly of steam condensate, exposing coiled wires to the media.

II. REVISIONS OF TENTATIVES

Tentative Method of Test for Manganese in Industrial Waters (**D 858 – 45 T**).[6]—The following revisions are recommended for inclusion in this method:

Section 2.—Delete the last sentence and substitute the following: "Colored ions or ions which form insoluble periodates may offer interference."

Section 3.—Add the following sentence to the end of this paragraph: "A wave length of 522 mμ should be used for photometric measurements."

Section 6 (c).—Change to read as follows: "Add 10 ml. of H_2SO_4 (sp. gr. 1.84) to each Erlenmeyer flask and boil until SO_3 fumes appear. Add HNO_3 (sp. gr. 1.42) until color disappears (usually a few drops will suffice). Cool the flasks and add 25 ml. of distilled water, 10 ml. of HNO_3 (sp. gr. 1.42) and 5 ml. of phosphoric acid (85 per cent) and hold at a gentle boil while adding slowly 0.3 g. of potassium periodate. Boil 30 min. for maximum color development."

Section 6 (d).—Change to read as follows by the addition of the italicized words and the deletion of those in brackets: "Rinse each flask into its corresponding Nessler tube with small, successive increments of distilled water, [and] dilute to the 50-ml. mark with distilled water, *and thoroughly mix.*"

Section 6 (e).—Add the following note: "Note 3.—Some laboratory distilled water contains sufficient organic matter to impair the intensity of color of a permanganate solution. This should be guarded against."

Section 8.—Change to read as follows: "Results by this method are considered precise to 0.03 ppm."

Tentative Methods of Test for Silica in Industrial Waters (**D 859 – 45 T**):[6] A number of minor editorial revisions and the following changes are recommended for inclusion in this method:

Section 1 (b).—Change to read as follows: "(*b*) the referee method, and the non-referee method A cover the gravimetric determination of total silica in water. Non-referee methods B and C cover the colorimetric determination of crystalloidal (noncolloidal) silica only.

Section 3.—Change to read as follows: "3. Use only reagents of the quality known as reagent grade."

Section 7 (a).—Change the second sentence to read as follows: "Evaporate the sample to dryness in a 200-ml.

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[6] 1946 Book of A.S.T.M. Standards, Part III-A.

platinum evaporating dish on a water bath."

Add the following three notes to this paragraph:

NOTE 1.—For an accuracy of 0.2 ppm. the sample volume should be at least 1 liter.

NOTE 2.—An acid-cured, glazed porcelain evaporating dish may be substituted for platinum.

NOTE 3.—With samples larger than 200 ml. a larger evaporating dish should not be used but portions of the originally measured sample should be added to the evaporating dish as the evaporation proceeds. During the evaporation of such large samples the contents of the dish must be protected against contamination from atmospheric dust by a suitable cover, such as a ribbed watch glass.

New Section.—Add a new Section 9 as follows and renumber the subsequent sections accordingly:

9. *Precision and Accuracy.*—The accuracy of the determination depends directly upon the accuracy of weighing, all necessary precautions having been observed. With an analytical balance having a sensitivity of 0.1 mg. the precision and accuracy should be of the order of 0.2 mg. of silica. If the sample size is at least one liter, the total silica can be determined with precision and accuracy of 0.2 ppm.

Section 12 (a).—Renumber as 13 and change the second sentence to read as follows: "Evaporate the sample in a 200-ml. platinum evaporating dish (Note 2) to a volume of 100 ml."

Add the same three notes as recommended above in Section 7 (*a*).

New Section.—Add the following new Section 15:

15. *Precision and Accuracy.*—The accuracy of the determination is essentially the accuracy of weighing, as for the referee method (see Section 9). The precision is equivalent to that of the referee method, 0.2 mg. of silica, but with samples having high salt concentrations incomplete solution of some salt in the mixed hydrochloric-perchloric acid may occur. If the silica residue appears to be clear, white and uncontaminated, it may be assumed that the result obtained is within 0.5 mg. of the true value. These weight limits of precision and accuracy may be converted to parts per million by consideration of the volume of the sample taken.

Section 15.—Renumber as 17 and add the following sentence: "It is particularly useful for the analysis of waters containing less than 10 ppm. of silica and is intended primarily as a rapid control method."

Section 17 (b).—Change to read as follows: " (*b*) *Hydrochloric Acid (1:1).*—Add one volume of HCl (sp. gr. 1.18) to one volume of distilled water."

New Section.—Add the following new Section 23:

23. *Precision and Accuracy.*—Precision of the order of 0.01 ppm. in the range from 0.02 to 2 ppm., and 0.1 ppm. in the range from 2 to 50 ppm., can be obtained with this method. The accuracy depends upon the type of water analyzed and the apparatus employed, but may be ascertained by periodic comparative determinations using the referee method (see Section 9).

Section 22.—Renumber as 25 and add the following sentence: "It is particularly useful for the analysis of waters containing less than 10 ppm. of silica and is intended primarily as a rapid control method."

Section 23 (a).—Renumber as 26 and insert "matched" before "Nessler."

Section 24 (a).—Renumber as 27 (*a*) and change to read as follows: "*Hydrochloric Acid (1:50).*—Add 1 volume of HCl (sp. gr. 1.18) to 50 volumes of distilled water."

Section 24 (b).—Change to read the same as the present Section 17 (*a*).

New Section.—Add the following new Section 29:

29. *Precision and Accuracy.*—Precision of the order of 0.1 ppm. in the range from 0 to 50 ppm. can be obtained with this method. The accuracy depends upon the type of water analyzed and the apparatus employed, but may be ascertained by periodic comparative determinations using the referee method.

III. ADOPTION OF TENTATIVE AS STANDARD

Tentative Recommended Practice for Sampling Boiler Water from Stationary Boilers (**D 860 – 45 T**).[6]—The committee recommends that this recommended practice be approved for reference to letter ballot of the Society for adoption as standard without revision.

IV. REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Method for Determination of the Hydroxide Ion in Industrial Waters (**D 514 – 41**).[6]—Committee D-19 recommends the immediate adoption of the following revisions in this standard and accordingly requests the necessary nine-tenths vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society:

Section 4 (d).—Delete the clause "If phosphate is absent."

Section 4 (e).—Delete this paragraph and note, which read as follows:

> (*e*) If phosphate is ***present***, two or three drops of the LaMotte purple solution shall be added and the sample shall then be titrated to the end point of this indicator with the 0.02 *N* acid. A comparison blank should be used if the operator is not familiar with the end point of this indicator. The contents of the flask shall be swirled steadily during the titration.
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> NOTE.—The LaMotte purple indicator is recommended when phosphates are present since in this case the phenolphthalein end point at a pH value of 8 fades rapidly due to the resolution of strontium phosphate. At the LaMotte purple end point above 9.3 this does not occur so readily. If LaMotte purple solution of the concentration employed as a pH indicator is used in place of the 1 per cent solution, 1 ml. will be required.

V. REVISION OF STANDARD AND REVERSION TO TENTATIVE

Standard Methods of Sampling Plant or Confined Waters for Industrial Uses (**D 510 – 41**).[6]—The committee recommends that these methods be revised as appended hereto,[7] and reverted to tentative.

The recommendations appearing in this report have been submitted to letter ballot of the committee.[8]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II, Editorial (E. P. Partridge, chairman) edited the proposed revisions and new proposed methods originating in the other subcommittees.

Section A on the A.S.T.M. Manual on Industrial Waters, referred to earlier in this report, was organized during the year. Topical outlines of each chapter contents were prepared by the assigned authors and were reviewed at a meeting of this section. They are currently being issued to Committee D-19 members for suggestions and approval. The preparation of the substance for each chapter will be started following letter ballot approval of the outlines by the committee.

Subcommittee III on Methods of Sampling (F. R. Owens, chairman) is now functioning under three sections: A on Sampling of Water, B on Sampling of Water-Formed Deposits, and a new section C on Sampling of Steam.

Section A originated the revisions of Standard Method D 510 and the recommendation that Tentative Recommended Practice D 860 be adopted as standard without change, as referred to earlier in this report. The section reported progress on the development of a specification for sampling equipment. This specification is intended to present design and composition requirements for metals used for nozzles, cooling and condensing coils, and for containers used in the transportation of samples.

Section B has undertaken extensive revisions of the Tentative Recommended Practice for Field Sampling of Water-Formed Deposits (D 887 – 46 T).[6] These revisions are intended to expand the present content and to offer more detailed procedures directed primarily toward the sampling of surfaces rather than the sampling of specific equipment.

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[7] These revised methods were accepted by the Society and appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

[8] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Section C reported that a draft of a proposed method for sampling of steam to include necessary sampling apparatus and procedure was in preparation.

Subcommittee IV on Methods of Analysis (A. A. Berk, chairman) is now functioning under four sections: A on Analytical Methods, B on Electrometric Methods, C on Special Methods, and a new section, D on Chemical Analysis of Water-Formed Deposits.

In addition to the revisions in the methods of test for manganese (D 858); silica (D 859); and hydroxide ion (D 514), referred to earlier in this report, Section A has under consideration the following subjects: revision of two existing methods, for total carbon dioxide and calculation of carbonate ions (D 513 – 43), and for total aluminum and aluminum ion (D 857 – 45 T) in industrial waters; as well as proposed new methods of test for determination of acidity and alkalinity; hardness; alkali metals in water; iron; dissolved gases such as chlorine, chloramine and ammonia; and nitrogenous substances (nitrates and nitrites).

Section A has also completed a Proposed Method for Determination of Total Solids in Industrial Waters of High Purity which is appended to this report as information.

Section B has in preparation methods of test for the determination of electrical conductivity of industrial waters. Committee E-1 on Methods of Testing during the past year issued the Tentative Method for Determination of the pH of Aqueous Solutions with the Glass Electrode (E 70 – 46 T).[6] This method will be applied for use in industrial waters by the development of necessary sampling procedures.

Section C sponsored the two new Tentative Methods for the Application of X-Ray Diffraction Methods to Water-Formed Deposits[5] and for the Detection of Iron Bacteria in Industrial Water,[5] which are referred to earlier in this report.

Section D, organized during the year, is operating with subsections on preparation, complete chemical analysis, and special tests. Each of the subsections is investigating the subject matter to be included in a proposed method.

Subcommittee V on Classification (L. K. Herndon, chairman).—Section A is continuing a study of the subject of the classification of water for industrial uses. Section B prepared the new Tentative Method for Reporting the Results of the Analysis of Water-Formed Deposits,[5] referred to earlier in this report.

Subcommittee VI on Methods of Testing V. V. Kendall, chairman).—The subsection on the NDHA Corrosion Tester prepared the Tentative Recommended Practice for Corrosion Tests in Industrial Waters,[5] referred to earlier in this report.

Committee D-19 has authorized that this subcommittee develop a proposed method for service tests of tubular materials in lines carrying liquids, steam, or gases, for the purpose of determining the corrosivity of the media and the relative corrodibility of the materials used.

This report has been submitted to letter ballot of the committee, which consists of 87 members; 74 members returned their ballots, of whom 73 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

R. E. HALL,
Secretary.

APPENDIX

PROPOSED METHOD FOR DETERMINATION OF TOTAL SOLIDS IN INDUSTRIAL WATERS OF HIGH PURITY[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method[2] covers the gravimetric determination of total nonvolatile solids in industrial waters of high purity, particularly in steam and in distilled or demineralized water.

Application

2. (*a*) This method is intended primarily for use with water containing not more than 5 ppm. of total solids, but may be applied to water of any purity when accurate values are required.

(*b*) The method should be employed only on samples taken in accordance with the Tentative Methods of Sampling Water for Industrial Uses (A.S.T.M. Designation: D 510)[3] and confined in sample containers that will not contaminate the sample.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Sample Containers.*—Sample containers having a capacity of approximately 20 liters (5 gal.) and a tightly fitting lid. Tubular connections of the same material (Note 1) as the body of the container shall be provided at the extreme bottom (Note 2) and at the "full" position on the side near the top.

Note 1.—Most waters of high purity exhibit a pH in the range from 6 to 9; for samples of such waters, containers of aluminum, stainless steel, or block tin may be used. The container shown in Fig. 1 is an aluminum milk can that is commercially available.

Note 2.—A supporting base, if not already present, is required to protect and to provide access to the bottom connection.

(*b*) *Evaporation Assembly.*—An evaporation assembly (Note 3) consisting of a platinum dish of about 200-ml. capacity (weight approximately 40 g.) supported on an electrically heated hot plate within an enclosed space, with provision for the introduction of the water sample into the platinum dish and of filtered and heated air over the platinum dish and or the removal from the enclosed space of air carrying moisture.

Note 3.—A convenient arrangement of an evaporation assembly is shown in Fig. 2. The enclosed space is formed by a glass bell jar supported on a transite base, the center of which is cut out to receive the electric hot plate. Through the top of the bell jar passes the sample inlet tube of glass tubing approximately 5 mm. in diameter; this extends downward to the top of the platinum dish. The electrical leads for the control circuit and the outlet for water vapor and air, a glass tube 10 to 15 mm. in diameter, also pass through the top of the bell jar. Heated air enters near the bottom of the bell jar through glass tubes that are constricted and bent in such a manner as to give a swirling motion to the air over the top of the evaporating dish. The input to the heater is regulated by means of a manually-operated rheostat.

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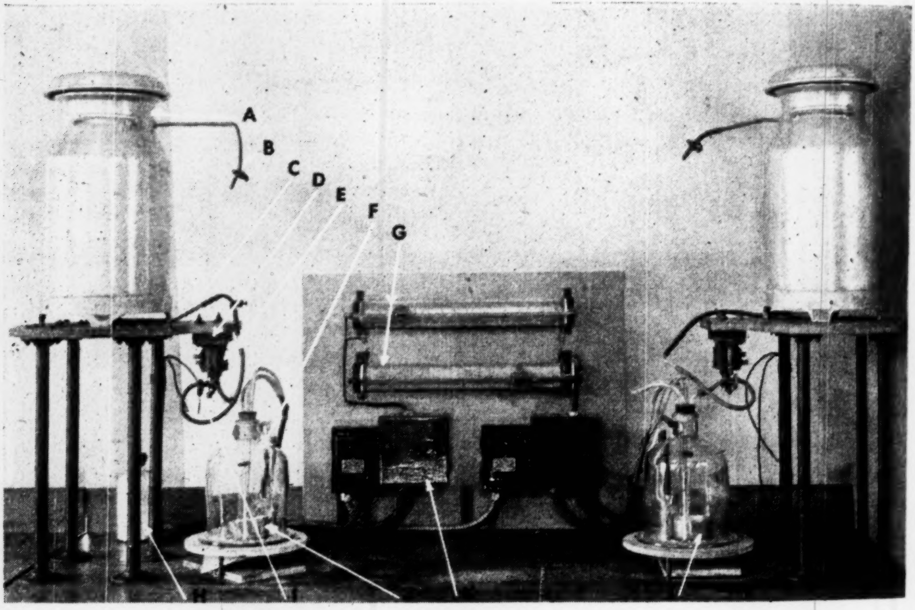
[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-19 on Water for Industrial Purposes. Published as information, June, 1947.

[2] For further information on this method, reference may be made to the paper by R. C. Ulmer, "Determination by the Evaporation Method of Small Amounts of Dissolved Solids in Water Such as Condensed Steam from Boilers," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 1221 (1939).

[3] 1947 Supplement to Book of A.S.T.M. Standards, Part III-A.

(*c*) *Air Purifier and Heater.*—An air purifier and heater in which the air is first passed through a filter to remove oil, water, and dust and then heated (Note 4).

NOTE 4.—The air purifier may consist of a 500-ml. or 1-liter leveling bottle filled with cotton batting and having a fine copper screen in the top to hold the cotton in the bottle. The air enters at the bottom. Although not generally required, for special uses a soda-lime tower may be included to remove carbon dioxide from the air stream. The air heater may consist of a heat-resistant glass tube,[4] approximately 15 mm. in inside diameter, wound with resistance wire in such a manner that the glass will not be heated above the softening range. About 60 watts will be required.



A—Sample container.
B—Overflow.
C—Filling tube of container.
D—Rubber shock absorber.
E—Solenoid (see Fig. 2).
F—Bell jar containing evaporation assembly (see Fig. 2).
G—Rheostats for adjusting rate of evaporation.
H—Air purifier.
I—Pinch clamp for regulating flow.
J—Platinum evaporating dishes.
K—Control assembly (see Fig. 3).

FIG. 1.—Complete Assembly of Apparatus for Evaporation of Steam Condensate.

(*d*) *Control Assembly.*—The wiring diagram of the control circuit is shown in Fig. 3, and indicates a satisfactory combination of specific component parts. The control assembly shall consist of the following:

(*1*) *Electrodes.*—Three platinum electrodes suspended in the evaporating dish at heights varying by about ¼ in. The top electrode shall be set at the height at which it is desired to maintain the water level (Note 5), that is, ½ to ¾ in. above the bottom.

(*2*) *Resistor.*—A resistor to limit the flow of current through the solution.

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[4] Pyrex glass is satisfactory for this purpose.

(3) *Relay.*—A relay to operate the heater and the solenoid controlling the flow of water.

(4) *Vacuum Tube.*—A vacuum tube to amplify the approximately 0.25-ma. current that flows through the solution in order that it will have sufficient force to operate the relay.

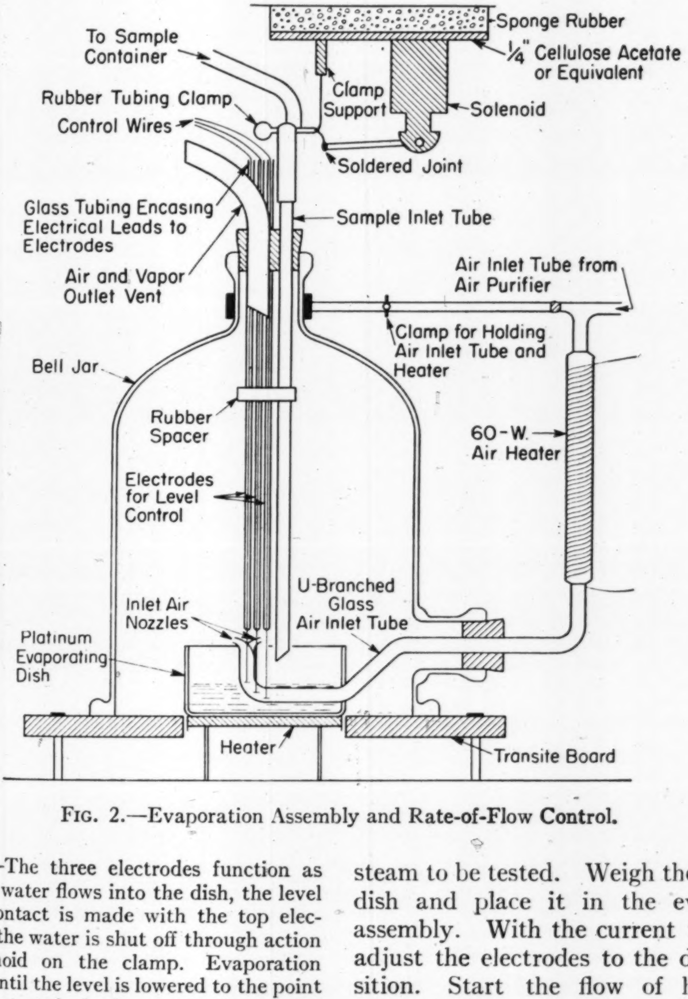

Sponge Rubber
To Sample Container
¼" Cellulose Acetate or Equivalent
Rubber Tubing Clamp
Clamp Support
Solenoid
Control Wires
Soldered Joint
Glass Tubing Encasing Electrical Leads to Electrodes
Sample Inlet Tube
Air and Vapor Outlet Vent
Air Inlet Tube from Air Purifier
Clamp for Holding Air Inlet Tube and Heater
Bell Jar
Rubber Spacer
60-W. Air Heater
Electrodes for Level Control
U-Branched Glass Air Inlet Tube
Inlet Air Nozzles
Platinum Evaporating Dish
Heater
Transite Board

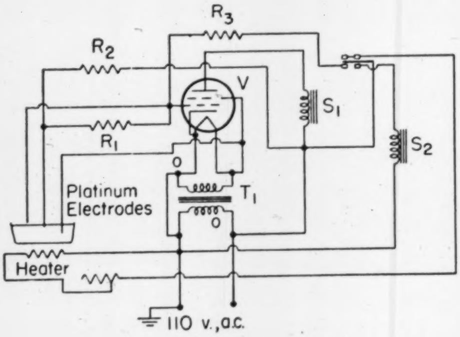
FIG. 2.—Evaporation Assembly and Rate-of-Flow Control.

NOTE 5.—The three electrodes function as follows. As water flows into the dish, the level rises until contact is made with the top electrode, when the water is shut off through action of the solenoid on the clamp. Evaporation takes place until the level is lowered to the point at which contact is broken with the middle electrode, when the solenoid opens the clamp allowing water to run in until the level again reaches the top electrode. When contact is broken with the bottom electrode, for example at the end of a run, current no longer flows to the heater, thus eliminating the danger of the dish becoming overheated. If the power fails, stopping the evaporation, the clamp, which normally is in the closed position, prevents running over of the dish.

Procedure

4. (*a*) Transfer to the sample container 20 liters of the water or condensed steam to be tested. Weigh the platinum dish and place it in the evaporation assembly. With the current turned off, adjust the electrodes to the desired position. Start the flow of heated air through the apparatus. Manually start the flow of water from the sample container into the dish by holding the clamp open, until the water just covers the top electrode. Turn on the current,

which closes the control circuit and by means of the relay supplies current to the heater under the dish. Adjust the rheostat on the heater so that the temperature of the water in the evaporating dish will be just below the boiling point. *Do not allow the water in the dish to boil.*



*R*₁—2.5-megohm resistance.
*R*₂—10-megohm resistance.
*R*₃—10-megohm resistance.
*S*₁—90 to 135-v. a-c. relay coil having a resistance of 590 ohms. (Marvel type QQ, manufactured by Specialties Mfg. Co., Detroit, Mich., or equivalent, is satisfactory.)
*S*₂—Solenoid for operating valve. (GE solenoid No. C.R. 9503, Size 2080, or equivalent, is satisfactory.)
*T*₁—6.3-v. filament transformer.
V—Type 2051 gas-filled tetrode (vacuum tube).
o—Phased as indicated.

NOTE: *Caution.*—For maximum tube life, all three electrodes must be immersed in water before this control is turned on.

FIG. 3.—Electronic Control Circuit.

NOTE 1.—During the starting-up period of about 15 min., it is well to observe the apparatus as "chattering" of the relay-solenoid combination sometimes takes place until the water in the dish becomes hot. If chattering occurs, the relay may be held closed manually until the water becomes hot.

(*b*) After the evaporation is complete (3 to 5 days of continuous operation) dry the dish and residue at 105 C. for 1 hr., cool, and weigh.

NOTE 2.—Care should be taken that the small amount of residue adhering to the electrodes is washed into the dish with the minimum amount of distilled water before the dish is dried. Care should also be taken to remove all foreign material that might adhere to the outside of the dish.

(*c*) If more than 1.0 ppm. of total solids is present in the water, a correspondingly smaller sample may be evaporated. The amount of residue to be weighed should be not less than 20 mg.

Calculation

5. Calculate the total solids present in the sample, in terms of parts per million, as follows:

$$\text{Total solids, ppm.} = \frac{w_2 - w_1}{v}$$

where:

w_1 = weight of platinum dish, in milligrams,
w_2 = weight of dish plus residue, in milligrams, and
v = liters of water evaporated.

REPORT OF COMMITTEE D-20*
ON
PLASTICS

Since the June, 1946, meeting in Buffalo, N. Y., Committee D-20 on Plastics has held two meetings; one in Atlantic City, N. J., in October, 1946, and the other in Cincinnati, Ohio, in March, 1947. The latter meeting was noteworthy in that a Symposium on Plastics[1] was held covering the general field of plastics with the following nine papers being presented:

"Permanence of Plastics," by G. M. Kline and F. W. Reinhart,

"Flammability of Plastics," by M. M. Braidech,

"The Electrical Properties of Plastics," by A. J. Warner,

"The Effect of Fungi and Humidity on Plastics," by J. Leutritz, Jr.,

"Low Pressure Moldings and Laminates," by T. W. Noble,

"The Mechanism of Plasticization in Plastics," by J. K. Craver,

"Plastics as Optical Materials," by H. R. Moulton,

"Metals and Plastics," by R. G. Chollar, and

"Automotive Glazing with Plastics," by George B. Watkins and J. D. Ryan.

The symposium was well attended by representatives of the various organizations and firms in the Cincinnati area as well as by the press. The symposium committee consisted of A. W. Downes (chairman), W. C. Goggin, and C. J. Frosch.

R. B. Rice has been appointed chairman of Subcommittee II on Hardness Properties to succeed L. Boor. E. B. Cooper has been appointed chairman of Subcommittee III on Thermal Properties to succeed W. A. Zinzow. G. W. Ingle has been appointed chairman of Subcommittee IV on Optical Properties to succeed W. F. Bartoe. F. W. Reinhart has been appointed chairman of Subcommittee VI on Specifications to succeed G. M. Kline. J. D. Ryan has been appointed chairman of Subcommittee VIII on Research to succeed H. K. Nason. F. L. Stiegler was appointed to represent Committee D-20 on Committee D-6 on Paper and Paper Products. G. M. Kline has been appointed to represent Committee D-20 on Committee E-1 on Methods of Testing.

The special joint advisory committee of D-9 on Electrical Insulating Materials andCommitteeD-20 to advise the armed forces in connection with the technical problems arising from the research program on plastics at Princeton University has accomplished its main task, namely, assistance in setting up the program and has had several meetings with the representatives of the armed forces and Princeton University.

The following revised scope of Committee D-20 was approved by the Board of Directors:

> *Scope.*—The development of test methods and specifications applicable to plastics and their raw materials, and to the finished products made therefrom (molding compounds, sheets, tubes and rods, and molded or fabricated articles). The scope does not include electrical tests, and materials used in the paint industry and rubber industry, which are covered by other committees.

It is with the deepest regret that Committee D-20 has to record the death of

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] To be issued as a separate publication in February, 1948.

Dr. R. J. Moore of the Bakelite Corporation, a staunch supporter of the work of the committee.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the proposed Tentative Definition of the Noun Plastic. This recommendation was accepted by the Standards Committee on July 10, 1946, and the new tentative appears in the 1946 Book of A.S.T.M. Standards, Part III-B, bearing the designation D 883 - 46 T.

On September 9, 1946, the Administrative Committee on Standards accepted the following recommendations submitted by Committee D-20:

Revision of Tentative Specifications for:

Cellulose Acetate Molding Compounds (D 706 - 44 T),
Laminated Thermosetting Materials (D 709 - 44 T),
Cellulose Acetate Plastic Sheets (D 786 - 45 T), and
Ethyl Cellulose Molding Powders (D 787 - 44).

The revised tentative specifications appear in the 1946 Book of A.S.T.M. Standards, Part III-B.

RECOMMENDATIONS AFFECTING STANDARDS

The committee plans to submit a number of recommendations to the Administrative Committee on Standards subsequent to the Annual Meeting. These recommendations have been submitted and approved by letter ballot of the committee, the results of which will be reported to the Standards Committee.[2]

ACTIVITIES OF SUBCOMMITTEES

The work of the subcommittees during the year has continued at a high level of effort, with the revised scope of the committee necessitating expansions in the activities of the Subcommittees on Specifications and Analytical Procedures. Sustained interest in the work of the Subcommittee on Research is reflected by the large attendances at the subcommittee meetings.

Subcommittee I on Strength Properties M. E. Marks, chairman).—A study of procedures for tension testing of rods and tubes is in process and work has been reported on a study of compression testing. Impact testing continues to necessitate considerable attention, and criticism of the existing Izod test is being studied. Some work on fatigue has been undertaken in conjunction with Committee D-9. Shear strength studies have advanced to the stage where a proposed method has been drawn up for submission to the subcommittee and a similar situation exists in regard to bearing strength.

Work on flexure has continued actively and agreement has been reached on the desirable span-depth ratio. Attention has been given to the effect of loading-edge radii, and the problem of testing thin specimens. Work on the Tentative Method of Test for Stiffness in Flexure of Nonrigid Plastics (D 747 - 43 T) has resulted in a proposed revision which has received the approval of the subcommittee.

Subcommittee II on Hardness Properties (R. B. Rice, chairman).—Work on a revision of the Rockwell hardness test has progressed to the stage of letter ballot of Committees D-9 and D-20. Interest has been expressed in a method for evaluating abrasion resistance, utilizing the Taber Abraser and round-robin tests have been initiated. Other methods of measuring hardness properties are under active consideration.

Subcommittee III on Thermal Properties (E. B. Cooper, chairman) has been very active, particularly in the field of flammability and flow of thermoplastic materials. In connection with the former, a detailed report of the work done by this section at North Carolina State College was given in a paper presented at the March, 1947, meeting of Subcommittee VIII by E. M. Schoenborn and D. S. Weaver, Jr.[3] A noteworthy round-robin test was conducted by L. W. A. Meyer in connection with the flow of thermoplastic materials as a result of which it has been recommended that no change be made in the tentative Method of Test for Measuring the Flow Properties of Thermoplastic Molding Materials (D 569 - 44 T). Work is under way on a revision of the Tentative Method for Heat Distortion Temperature of Plastics (D 648 - 45 T). A revision of the Tentative Method of Test for Measuring the Relative Mobility of Thermosetting Molding Materials (D 731 - 44 T) has been sent to letter ballot of Committee D-20.

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[2] See Editorial Note, p. 424.

Subcommittee IV on Optical Properties (G. W. Ingle, chairman).—Work continues on the problems associated with light transmission. A tentative method, using the sphere-hazemeter is ready for subcommittee approval. In addition to the sphere-hazemeter and the General Electric Spectrophotometer, the Hunter Null-method Hazemeter is being reviewed for measuring haze.

Section V on Permanence Properties (J. H. Teeple, chairman).—Consideration has been given to the applicability to plastics of the Tentative Recommended Practice for Operating Light and Water Exposure Apparatus (Carbon-Arc Type) for Testing Paint, Varnish, Lacquer and Related Products (D 822 - 46 T), prepared by Committee D-1. This recommended practice has now been approved by letter ballot of Committee D-20. Further work is planned on correlations between results obtained with the above equipment and those obtained by outdoor exposure tests. Studies of a Proposed Method of Test for Weight Loss of Plastics on Heating, using the Abderhalden equipment have resulted in the method being submitted to letter ballot of Committee D-20.[2] Work continues on studies in the field of moisture-vapor transmission, and in methods of measuring shrinkage. A revision of the Tentative Methods of Test for Resistance of Plastics to Accelerated Service Conditions (D 756 - 46 T) has been accepted by letter ballot of Committee D-20.

Subcommittee VI on Specifications (F. W. Reinhart, chairman).—Very active work has been pursued in this section, especially in the new assignment of work on specifications for plasticizers. A. W. Downes has been appointed chairman of this section and a large group is engaged in consideration of the individual materials with emphasis on the chemical purities thereof. This section will therefore coordinate its work closely with Subcommittee VII.

Subcommittee VII on Analytical Methods (P. D. Brossman, chairman).—This subcommittee, like Subcommittee VI, has had the scope of its work extended by the consideration of plasticizers, and a new section has been set up to handle the work. It is currently engaged in determining what tests are considered necessary in this field. A method for the determination of free phenol and free formaldehyde in phenol-formaldehyde resins has been actively worked upon. A Method of Test for Specific Gravity of Plastics has been completed and has received the approval of Committee D-20 by letter ballot. Results of round-robin tests on the determination of plasticizer in cellulose ester plastics has shown the need for a revision of this test method and work to accomplish this is under way.

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[3] E. M. Schoenborn and D .S. Weaver, Jr., "Ignition Temperatures of Rigid Plastics," ASTM BULLETIN, No. 146, May, 1947, p. 80.

Subcommittee VIII on Research (J. D. Ryan, chairman) is paying particular attention to the fundamental aspects of impact testing and flow properties. Papers of an original nature have been presented by various members. A full discussion has taken place on the merits and demerits of promoting work of an investigational nature to be carried out in the laboratories of academic institutions. It was agreed that such development programs as are already in existence should be followed by the subcommittee in an advisory capacity through its membership as in the past, but that no fund-raising activities for the purpose of activating programs be entered into.

Subcommittee IX on Molds and Molding (L. A. Sontag, chairman).—Work in the subcommittee has continued on molding procedures for thermosetting molding compounds and a revision of the Tentative Recommended Practice D 796 - 45 T has been approved by letter ballot of the subcommittee. A proposed method for measuring shrinkages of molded plastics is being actively worked on and the proposal submitted to the subcommittee for approval. Especial attention is being given to the problem of working out a standardized mold for the preparation of test specimens of thermoplastic materials.

Subcommittee X on Definitions, Nomenclature, and Significance of Tests (G. W. Clark, chairman).—Many statements on significance of tests are under active review by the subcommittee. A proposed standard system for designating the direction of testing specimens has been worked out and is being circulated for comment. Continued work is being undertaken on the definitions of terms such as plastic (as a noun), foil, film, and sheet.

This report has been submitted to letter ballot of the committee, which consists of 159 members, 54 members returned their ballots of whom 39 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ROBERT BURNS
Chairman.

A. J. WARNER,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentatives:

- Method of Test for Weight Loss of Plastics on Heating (D 948 - 47 T), and
- Recommended Practice for Molding Specimens of Phenolic Materials for Use in Electrical Tests (D 949 - 47 T).

Revision of Tentative Specifications for:

- Cellulose Acetate Molding Compounds (D 706 - 46 T), and
- Cellulose Acetate Butyrate Molding Compounds (D 707 - 46 T).

These recommendations were accepted by the Standards Committee on September 4, 1947, and the new and revised tentatives appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part III-B.

REPORT OF COMMITTEE E-1*
ON
METHODS OF TESTING

Committee E-1 on Methods of Testing, through its Advisory Committee, has given consideration during the year, at the request of the Society's Board of Directors, to a proposed reorganization of the committee in order that it might function more effectively in view of the expansion in activities of A.S.T.M. technical committees which has resulted in newer types of testing problems. The conclusions reached following subsequent considerations of the matter by the Board of Directors are given in the Report of the Board.[1] Briefly, it is proposed to make the present E-1 Advisory Committee an administrative group and to increase the number of members from nine to twelve, appointed by the Board of Directors for terms of three years each. It is also proposed to discontinue the present "Representative Members" on the main committee, but to continue and enlarge the number of representative members serving on the E-1 sections. In place of the present technical committees, advisory groups will be established on particular subjects, and each advisory group will determine and appoint whatever sections or task groups are necessary. This plan contemplates review of the internal committee structure every two years.

Since one of the principal functions of Committee E-1 is to review methods prepared by other Society committees, it was recommended that technical committees should refer new and revised methods and specifications containing requirements for apparatus to Committee E-1 for review, preferably while in draft form and before presentation to the Society as tentative, but in any case prior to their reference to the Society for adoption as standard. Further, reports to the Society on recommendations affecting methods of test shall state whether the method has been referred to Committee E-1 for review and what action has been taken by Committee E-1.

In line with this reorganization, the Board of Directors has appointed to the E-1 Advisory Committee Messrs. R. L. Templin, F. D. Tuemmler, and J. R. Townsend, for terms of one, two, and three years, respectively, and has reappointed Messrs. W. H. Fulweiler, H. F. Moore, and T. S. Fuller each for another term of three years.

A number of the technical committees and sections of Committee E-1 have been active during the year in reviewing standards prepared by other Society committees and also in the revision of several standards under the jurisdiction of Committee E-1. These matters were considered at meetings of the E-1 subgroups as noted later in this report.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1946 Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] See p. 41.

New Tentative Method for:

Determination of the pH of Aqueous Solutions with the Glass Electrode (E 70 - 46 T).

Revision of Tentative Specifications for:

ASTM Thermometers (E 1 - 46 T), comprising specifications for nine new thermometers and in addition changes in certain fractions of the existing specifications.

These recommendations were accepted by the Standards Committee on December 5, 1946, and the new and revised tentatives appear in the 1946 Book of A.S.T.M. Standards, Parts II, III-A, and III-B.

On December 5, 1946, the Administrative Committee on Standards accepted proposed revisions in the Tentative Methods of Compression Testing of Metallic Materials (E 9 - 33 T). The revised tentative methods, bearing the designation E 9 - 46 T, appear in the 1946 Book of A.S.T.M. Standards, Parts I-A, and I-B.

I. ADOPTION OF TENTATIVE AS STANDARD

On the recommendation of the Section on Indentation Hardness (J. R. Townsend, chairman), it is proposed that the Tentative Hardness Conversion Tables for Steel (Relationship Between Diamond Pyramid Hardness, Rockwell Hardness, and Brinell Hardness) (E 48 - 43 T) be approved for reference to letter ballot of the Society for adoption as standard. These conversion tables were quite extensively used during the war and the committee has received no adverse comment or criticism concerning them. It is accordingly felt that they should be formally approved as standard.

II. REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends that the Standard Specifications for A.S.T.M. Thermometers (E 1 - 46) be revised by changing the lower range of the Centigrade thermometer 6C from "−60 C." to read "−80 C." and in the Fahrenheit thermometer 6F from "−70 F." to read "−112 F." This revision is in accordance with the revision in the Standard Method of Test for Cloud and Pour Points (D 97 - 39) recommended by Committee D-2 on Petroleum Products and Lubricants.

III. EDITORIAL CHANGES IN STANDARD

Also on the recommendation of the Section on Indentation Hardness, the following editorial changes are proposed in the Standard Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 - 42):

Section 1.—In the note following this section, change the third sentence to read as follows by the addition of the italicized words: "The hardness value, as read from the dial, is an arbitrary number which is related to the depth of indentation *caused by two superimposed impressions*, and since the scales are reversed, the number is higher the harder the material."

In this same note, add the following new sentence following the present fifth sentence: "This major load is the total load applied and the depth measurement depends solely on the increase in depth due to increase from minor to major load."

Section 8.—Change the third paragraph of the note immediately preceding this section to read as follows by the addition of the italicized words: "The minor load applied by the Rockwell superficial hardness tester is 3 kg. The major load (*total load*) is 15 kg., 30 kg., or 45 kg."

ACTIVITIES OF TECHNICAL COMMITTEES

Technical Committee I on Mechanical Testing (H. F. Moore, chairman):

This technical committee has been active through its various sections, five of which met during the year, as mentioned in the following brief summary.

Section on Tension Testing (R. L. Templin, chairman).—At a meeting of this section held in Philadelphia, Pa., on February 25, 1947, the committee reviewed the Standard Methods of Tension Testing of Metallic Materials (E 8 - 46) which had been revised in 1946 to include a number of changes proposed by Committee A-1 on Steel. In addition, the section on speed of testing was revised in line with the recommendations from the E-1 Section on Effect of Speed of Testing. No further changes in Standard E 8 were proposed at the meeting.

The section also received a progress report from the special committee appointed to review the various A.S.T.M. methods of tension testing of nonmetallic materials. This review of the present methods is continuing and a further report will be made at a later date.

The section has reviewed by correspondence a proposed Tentative Method for Bearing Strength Test of Plastics, prepared by Committee D-20 on Plastics. A number of comments and suggestions received from the members of the section on this proposed new method have been forwarded for consideration to the sponsoring committee.

Section on Compression Testing (M. F. Sayre, chairman).—At a meeting of this section held in Atlantic City, N. J., on November 18, 1946, a revision of the Tentative Methods of Compression Testing of Metallic Materials (E 9 - 33 T) was completed and was submitted through the Standards Committee as mentioned earlier in this report. The section also reviewed at that time the first draft of the Proposed Tentative Methods of Compression Testing of Metallic Materials in Sheet Form, which had been prepared by a special subcommittee. A number of changes were proposed at this meeting which will be incorporated in the revised method to be considered at the meeting of the section to be held during this Annual Meeting.

Section on Impact Testing (W. W. Werring, chairman).—This section has been giving a very thorough review to the Tentative Methods of Impact Testing of Metallic Materials (E 23 - 41 T). At a meeting held in Buffalo, N. Y., on June 25, 1946, attention was given to criticisms received, particularly as concerns the matter of notch effects and "true impact." Based on the revisions discussed at this meeting, the chairman prepared a proposed revision of Methods E 23 which were later reviewed in detail at a meeting in Philadelphia, on February 26, 1947. After a full discussion of the proposed changes, it was decided that further study of the revision was desirable and the matter accordingly will receive consideration at the June, 1947, meeting prior to submission to the Society.[2]

Section on Effect of Speed of Testing (P. G. McVetty, chairman).—The proposed revision of the paragraph on speed of testing proposed by this section was included in the revised Standard Methods of Tension Testing of Metallic Materials (E 8 - 46). At a meeting in Philadelphia, Pa., on February 25, 1947, these provisions were further reviewed and it was decided that no further change should be made at this time.

The section is sponsoring a Round-Table Discussion on Speed of Testing to be held during this Annual Meeting on June 18, 1947. This informal discussion is a preliminary step looking toward the sponsoring of a symposium on this subject, possibly at the 1948 Annual Meeting. The main purpose of the round-table discussion will be to consider undesirable features in existing speed of testing requirements now appearing in product specifications and possibly to find procedures for correcting them.

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[2] See Editorial Note, p. 430.

A Bibliography on Speed of Testing recently compiled by a subgroup will bring up to date the former lists prepared in 1936 and 1938.

Another subgroup has in preparation a report on the possibilities and limitations of existing testing equipment with respect to control of speed of testing.

Section on Elastic Strength of Materials (F. B. Seely, chairman).—At its meeting in June, 1946, in Buffalo, N. Y., this section completed a number of editorial changes in the Standard Definitions of Terms Relating to Methods of Testing (E 6 - 36) in line with the revisions approved last year in the revised Methods of Tension Testing (E 8) and Compression Testing (E 9).

Section on Calibration of Testing Machines (H. F. Moore, chairman)—The section has completed one of the projects mentioned in the 1946 Report, namely, the development as separate standards of the Methods of Verification of Testing Machines, and the Methods of Verification of Calibration Devices for Verifying Testing Machines. Following meetings of the section held in Buffalo, N. Y., in June, 1946, and in Philadelphia, Pa., on February 26, 1947, the committee completed and approved by letter ballot these two new tentative methods which will replace the present Standard Methods of Verification of Testing Machines (E 4 - 36). It is expected that the two new tentative methods will be submitted to the Administrative Committee on Standards subsequent to the Annual Meeting.[2] Separate methods were considered necessary since the requirements are more strict for calibrating apparatus than for testing machines and the single standard had caused some confusion.

Technical Committee II on Consistency, Plasticity, and Related Properties (W. F. Fair, Jr., chairman):

This technical committee has under review the present A.S.T.M. methods of test for consistency, flow, softening point, etc. When this review has been completed, the suggested comments for improving these methods will be presented for consideration to the sponsoring technical committee.

The Section on Methods and Apparatus for Absolute Viscosity Measurements (W. F. Fair, Jr., chairman) is continuing to compile descriptions of methods and apparatus used for determining absolute viscosity. The section has decided to include in this description of absolute viscometers those which have a sound theoretical background and are capable of yielding results that permit calculation of shearing stresses, rates of shear and viscosity all in absolute units; that is, in dynes per square centimeter, reciprocal seconds and poises, respectively.

The Section on Softening Point (W. A. Kirklin, chairman) has in preparation a draft of methods of ring and ball softening point test which would comprise a consolidation of the present A.S.T.M. Ring-and-Ball Method (D 36) and the Ball and Shouldered Ring Apparatus (E 28).

Technical Committee III on Particle Size and Shape (L. T. Work, chairman):

The Section on Pigment-Type Materials (C. E. Barnett, chairman) has circulated among its members a questionnaire to determine what methods are being used for particle size measurements since there were a number of developments in this testing field during the war. Based on the information received from this questionnaire, the section has decided to establish task groups on individual methods of particle size measurement. As a start in this direction, three such task groups are being organized on the electron microscope, optical microscope, and gas adsorption methods. At a later date consideration will be given to the establishment of similar task groups on other methods such as by liquid adsorption, permeability, liquid sedimentation (gravity and centrifugal methods), air classification, and turbidity or light scattering. The Task Group on Optical Microscope (R. P. Loveland, chairman) will undertake a revision of the Tentative Method of Test for Particle Size Distribution of Subsieve Size Particulate Substances (E 20 – 33 T).

Technical Committee IV on Methods for Density (W. Souder, chairman):

This technical committee has been without a chairman since the resignation of Mr. H. W. Bearce following his retirement from the National Bureau of Standards. The newly appointed chairman, Mr. Wilmer Souder, Chief of the Metrology Division at the National Bureau of Standards, is proceeding with plans for a review of the various methods for density, specific gravity, and related properties now published by the Society. In response to a request received, this technical committee is undertaking the preparation of a method of determining the apparent or bulk density of powdered chemicals and drugs (pharmaceuticals).

Technical Committee X on Conditioning and Weathering (Robert Burns, chairman):

This technical committee has had under consideration a change in the temperature for standard laboratory atmosphere from 25 C. (77 F.) to a value of 24 C. (75 F.) at a relative humidity of 50 ± 2 per cent, in order to obtain a greater degree of uniformity since certain materials are now required to be tested at temperatures slightly different from 25 C. (77 F.) but at 50 per cent relative humidity. The committee considered this matter at its meeting on February 25, 1947, in Philadelphia, Pa., but before reaching a final decision is awaiting consideration of the matter by several Society committees.

The technical committee has recently been advised that the Federal Specifications Board's Technical Committee on Standard Temperatures and Relative Humidity Conditions has recommended that the following standard laboratory atmosphere conditions be adopted for procurement testing under Federal Specifications: 73.5 F. (± 2 F. unless otherwise specified) and 50 per cent relative humidity (± 2 per cent unless otherwise specified), the standard conditions to become effective after a conversion period of one year.

The technical committee will review this entire matter at its next meeting to be held in the Fall of 1947.

Technical Committee XII on Laboratory Apparatus (W. H. Fulweiler, chairman):

Section A on Thermometers (R. M. Wilhelm, chairman) held three meetings during the year, one last June in Buffalo, N. Y., and two in Philadelphia, Pa. on January 7, and April 25, 1947, and has continued its program of developing new specifications for thermometers as requested by other Society committees. It has undertaken a detailed editorial review of the existing thermometer specifications. New specifications for a Stormer viscosimeter thermometer requested by Committee D-17 on Naval Stores, and for a thermometer for the oil content test of paraffin wax requested by Committee D-2 have been completed and will be submitted as tentative through the Standards Committee.[2]

The second draft of the Proposed Method of Testing and Standardization of Liquid-in-Glass Thermometers is under review. The committee has also received a request from several committees for new thermometers for use in testing bulk shipments of petroleum, oxidation tests of turbine oil, oxidation stability of lubrication greases, and for butadiene content.

Section C on Volumetric Glassware (J. J. Moran, chairman) reviewed the glassware apparatus prescribed in new methods submitted during the past year, which resulted in the adoption of certain immediate changes and others are under consideration by the technical committees sponsoring such methods.

Technical Committee XIII on Hydrogen Ion Determinations (E. B. Ashcraft chairman):

The new Tentative Method for the Determination of the pH of Aqueous Solutions with the Glass Electrode (E 70 - 46 T) mentioned earlier in this report was prepared by this technical committee.

The Symposium on pH Measurement held at the 1946 Annual Meeting was sponsored by this technical committee. The symposium will shortly be issued as a separate publication.[3]

This report has been submitted to letter ballott of the committee, which consists of 12 members; 12 members returned their ballots, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

W. H. FULWEILER,
Chairman.

P. J. SMITH,
Ex-officio Secretary.

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[3] Available as a separate publication, July, 1947.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods of:

Verification of Calibration Devices for Verifying Testing Machines (E 74 - 47 T).

Revision of Tentative Specifications for:

A.S.T.M. Thermometers (E 1 - 46 T).

Revision and Reversion to Tentative of:

Methods of Verification of Testing Machines (E 4 - 36).

These recommendations were accepted by the Standards Committee on November 6, 1947 and appear in the 1947 Supplement to Book of A.S.T.M. Standards; E 74 and E 4 in Parts I-A, I-B, II, III-A, and III-B, and E 1 in Parts II, III-A, and III-B.

On February 16, 1948, the Administrative Committee on Standards accepted proposed revisions in the Tentative Methods of Impact Testing of Metallic Materials (E 23 - 41 T). The revised tentative methods appear in the 1947 Supplement to Book of A.S.T.M. Standards, Part I-A, bearing the designation E 23 - 47 T.

REPORT OF COMMITTEE E-3*

ON

CHEMICAL ANALYSIS OF METALS

Committee E-3 on Chemical Analysis of Metals has held one meeting during the year, at Buffalo, N. Y., on June 26, 1946. This cannot be taken as a true measure of the accomplishments of the committee because its work is done largely by its divisions and subcommittees, and these have been most active. A new edition of the Book of A.S.T.M. Methods of Chemical Analysis of Metals was issued in the summer of 1946, and this was the culmination of intensive work by members of Committee E-3 during the previous year. Subcommittees have already begun to review procedures in preparation for providing up-to-date methods for the next edition of the Book. This work is not spectacular, and its results only come to light when the finished method is issued.

The Advisory Committee met twice, first on June 27, 1946, in Buffalo, N. Y., and on February 25, 1947, in Philadelphia, Pa. Matters of policy were carefully considered and plans for the future direction of the work of the committee were made.

Twenty divisional and subcommittee meetings were held at Buffalo and Philadelphia. In addition, special committee and group meetings were held to consider specific problems.

Ten new members were added to the committee during the year. Two committee members resigned. One member died. The active membership of the committee is now 91.

At the direction of the Advisory Committee, an editorial committee has been working on the development of recommendations on form to be used as a guide in writing analytical procedures.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the Annual Meeting, Committee E-3 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of:

Sampling Slab Zinc (E 65 – 46 T), and

Revision of Tentative Methods of:

Chemical Analysis of Lead- and Tin-Base Solder Metal (E 46 – 46 T).

These recommendations were accepted by the Standards Committee on July 10, 1946, and the new and revised tentatives appear in the 1946 Book of A.S.T.M. Methods of Chemical Analysis of Metals, pp. 260 and 232.

I. NEW TENTATIVE METHOD

Committee E-3 recommends that the proposed Photometric Method for Determination of Boron in Steel[1] be accepted for publication as tentative.

II. ADOPTION OF TENTATIVES AS STANDARD

Committee E-3 recommends that the Tentative Methods of Chemical Analysis of Steel for Sulfur by Direct Combustion and for Nitrogen (E 30 - 46 T)[2] and the Tentative Methods of Chemical Analysis of Ferro-Alloys (E 31 - 46 T)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] See Editorial Note, p. 434.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

Two tentatives under the jurisdiction of Committee E-3 have stood for more than two years without revision. They are the Tentative Methods of Chemical Analysis of Metallic Materials for Electrical Heating (E 38 - 42 T) and Method for Sampling Molybdenum Salts and Compounds for Metallurgical Use (E 66 - 34 T). In the intensive work of revision of procedures in preparation for the 1946 Book of A.S.T.M. Methods of Chemical Analysis of Metals, priority was given to methods other than those mentioned above. In the near future consideration will be given to the status of these two tentatives.

ACTIVITIES OF DIVISIONS AND SUBCOMMITTEES

Division A on Ferrous Metals (Arba Thomas, chairman) held two meetings during the year, one at Buffalo, N.Y., in June, 1946, and at Philadelphia, Pa., in February, 1947. Both meetings were all-day sessions and very well attended with keen interest shown by all present.

The work on the Photometric Method for Determination of Boron in Steel was completed and submitted to Committee E-3 for letter ballot. There were two negative votes cast, because of certain minor disagreements regarding the manner in which the method was written. The procedure has been rewritten to overcome these objections.

The subgroup responsible for the development of a method for the determination of tin in alloy steels has completed its work, and as soon as this procedure is rewritten with certain editorial revisions, it will be submitted to the Division for letter ballot.

Considerable work has been carried on by the subgroup in charge of the development of a method for the determination of aluminum in alloy steels. This work has been in charge of C. Ruhe and has been confined to a complex highly alloyed steel. At the February, 1947, meeting of the Division it was decided to make this procedure applicable to steels of the stainless type only. It was also decided to do some further work covering a wider range of aluminum content, preferably from approximately 0.03 to 0.20 per cent. E. J. Dunn agreed to work with Mr. Ruhe on this matter and secure for him samples of stainless steel carrying approximately 0.03 and 0.15 per cent aluminum. As soon as the cooperative analytical work is completed on these additional samples, this method will be ready for letter ballot by the Division.

The subgroup headed by T. R. Cunningham in charge of the development of a method for the determination of beryllium in alloys of the turballoy type reported that considerable work had been done and that in most instances the cooperating analysts had obtained very satisfactory beryllium values using the proposed procedure. One member of the subgroup who obtained unsatisfactory values will be requested to recheck his work. When this checking has been completed, the method will be written up in standard form and submitted to the Division for letter ballot.

In an effort to overcome certain criticisms of the present published analytical methods for ferrous materials and to take advantage of new and modern techniques, the Division decided to review carefully all the published methods prior to the issuance of the next edition of the Book of Chemical Methods. The object of this detailed review is (*1*) to determine whether the published methods can be modified so as to improve the procedures from a time-saving and technique-simplification standpoint without sacrificing their present accuracy, (*2*) to determine whether there are other methods utilizing modern equipment and techniques and having, at least, the same degree of accuracy as the present published methods which can be substituted for the present methods, and (*3*) to study the performance of the proposed modified or entirely new methods on standard samples of ferrous materials with the object of obtaining analytical data to support the proposed modifications of the present published methods or the entirely new procedures.

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[2] 1946 Book of A.S.T.M. Methods of Chemical Analysis of Metals.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

To carry on this work, the chairman appointed 12 subgroup chairmen who are responsible for reviewing certain procedures. The chairmen selected for these subgroups were men who have been in the past the most active, and they were requested to select their own subgroup personnel. It was hoped that by setting up these subgroups, members of the Division who have not been so active would be included, thus stimulating their interest.

As the demand for analytical methods applicable to high-temperature alloys of the jet and gas-turbine types is increasing, it was deemed advisable by the Division at the February, 1947, meeting to begin development of methods for the analysis of these complicated alloys. R. M. Fowler was appointed chairman of a permanent subcommittee to head this work.

At the February meeting considerable discussion was held relative to the explosion and fire hazards of perchloric acid and hood and ductwork accumulations of this acid and its salts. Serious explosions and fires have occurred in many laboratories since the advent of this valuable and essential laboratory reagent, and it was the concensus of the Division that all A.S.T.M. published analytical methods wherein the use of perchloric acid was specified should carry statements regarding its hazards.

Division B on Non-Ferrous Metals (D. R. Evans, chairman) held two meetings during the year, one at Buffalo in June, 1946, and the other at Philadelphia in February, 1947. The activity of the members of this Division is predominantly expressed in the work of the several subcommittees, practically all of which held meetings at both Buffalo and Philadelphia. A new Subcommittee B-8 on Polarographic Methods was organized during the year under the chairmanship of W. W. Braun. At the suggestion of Subcommittee II of Committee B-9 on Metal Powders and Metal Powder Products, a subcommittee is in the process of formation with the coöperation of Division A to be concerned with the Analysis of Powdered Metals.

Intensive work is under way in the subcommittees on reviewing present procedures and developing new procedures looking forward to the next edition of the Book of Chemical Methods.

C. L. Luke was appointed chairman of Subcommittee B-2 on the Analysis of Lead, Tin, Antimony, Bismuth, and Their Alloys.

Division C on Sampling (W. C. Bowden, Jr., chairman) held one meeting during the year at Buffalo in June, 1946. It has been occupied with revising and editing many sampling procedures. Sixteen such revisions are now completed and have been distributed to the members for letter ballot. These revisions are partly editorial in order to establish a standard form and partly procedural.

At the request of Subcommittee II of Committee B-2 on Non-Ferrous Metals and Alloys, Division C is cooperating with it in revising the sampling procedure in the Specifications for Pig Lead (B 29 – 43). Subcommittee II desires that the method of sapling by punching be discarded and that procedures for kettle sampling and sampling for spectrographic examination be included.

The Division desires to include methods of sampling for spectrographic purposes on many metals and alloys and has approached Committee E-2 on Spectrographic Analysis for advice on this subject.

Division D on General Analytical Methods (S. E. Q. Ashley, chairman) held three meetings during the year, one at Buffalo in June, 1946, one at Philadelphia in February, 1947, and a Symposium on Precision and Accuracy, at the National Bureau of Standards in Washington, D. C., in October, 1946. Subcommittee D-1 on the Development, Preparation, or Standardization of Reagents is cooperating with the Committee on Analytical Reagents of the American Chemical Society. W. M. Murray, Jr., who has been chairman of this subcommittee, has resigned because of the pressure of this work and T. C. Bryson has been appointed to succeed him.

Subcommittees D-1 and D-2 are jointly responsible for the Tentative Recommended Practices for Apparatus and Reagents for Chemical Analysis (E 50 – 46 T) and are reviewing this section to keep it up to date.

Subcommittee D-2 is studying standards for filter paper, analytical balances, volumetric glassware, and for certain types of electrometric equipment including polarographic apparatus and equipment for dead-stop end point titrations.

J. H. Harley has been appointed chairman of Subcommittee D-3.

Subcommittee D-4 on Precision and Accuracy of Methods for the Analysis of Metals sponsored the symposium held in Washington and has drawn up a procedure to be followed in conducting cooperative tests so that the results will be suitable for statistical analysis.

This report has been submitted to letter ballot of the committee which consists of 91 members; 80 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. E. F. LUNDELL,
Chairman.

J. W. STILLMAN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-3 presented to the Society through the Administrative Committee on Standards the recommendation that the Photometric Method for Determination of Boron in Steel be accepted for publication as tentative. This recommendation was accepted by the Standards Committee on March, 1948, and the new tentative method appears as a separate reprint bearing the designation E 30 – 48 T.

REPORT OF COMMITTEE E-4*
ON
METALLOGRAPHY

Committee E-4 on Metallography has not had a meeting since that held during the 1946 Annual Meeting. During the past year several of the subcommittees have been doing the initial work on certain new proposals. However, the overall activity of Committee E-4 has not been great, due to the fact that all of the pending work was completed last year.

The membership changes during the year have resulted in a total of 80 members as of this date.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Selection and Preparation of Samples (J. R. Vilella, chairman).—Having revised the Tentative Methods of Preparation of Metallographic Specimens (E 3- 46 T) last year, there has been no activity since the last report.

Subcommittee VI on X-Ray Methods (W. L. Fink, chairman) is currently completing the drafts for proposals covering methods for the determination of preferred orientation in worked metals and for the determination of crystal orientation by the back reflection method. These proposals are expected to be ready for letter ballot in the immediate future.

In addition to the above, representatives of this group and of Committee E-4 have been cooperating with the American Society for X-ray and Electron Diffraction through the Joint Committee. Through these efforts, improved data cards and indexing will soon be available to users of the Hanawalt Method (E 43 46 T).

Subcommittee VIII on Grain Size (M. A. Grossman, chairman).—Since the Classification of Austenitic Grain Size in Steels (E 19 - 46) was adopted as standard last year, the Group on Austenitic Grain Size has had no further activity.

The Group on Ferrite Grain Size, R. E. Penrod, chairman, has held one meeting and is currently formulating suggestions outlining the work ahead.

In the Group on Non-ferrous Grain Size, C. H. Samans, chairman, the old non-ferrous standard grain size chart and method in Standard E 2 are being critically reviewed in the light of many comments and some criticism of this standard. In addition, a proposal for magnesium grain size is ready for formal action, and a companion method for aluminum is nearing completion.

Subcommittee IX on Inclusions (Samuel Epstein, chairman) did not meet, but during the year agreement was reached by correspondence with S.A.E. to correct a discrepancy between the S.A.E. Recommended Practice and the A.S. T.M. Recommended Practices for Determining the Inclusion Content of Steel (E 45 - 46 T). The S.A.E. has changed the definition of average length so that its recommended practice is now in complete agreement with Recommended Practices E 45.

This report has been submitted to letter ballot of the committee, which consists of 80 voting members; 52 members returned their ballots, of whom 49 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

MARY NORTON,
Secretary.

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* Presented at the Fiftieth Annual Meeting, June 16–20, 1947.

REPORT OF COMMITTEE E-5*

ON

FIRE TESTS OF MATERIALS AND CONSTRUCTION

Committee E-5 on Fire Tests of Materials and Construction, formerly known as Committee C-5, has held two meetings during the past year, one in Buffalo, N. Y., on June 17, 1946, and the other in Washington, D. C., on April 17, 1947. In conjunction with the latter, meetings were held of five of the six subcommittees.

The committee now consists of 52 members of whom 21 are classified as producers, 10 as consumers, 14 as general interest members, and 7 as consulting members.

I. ADOPTION OF TENTATIVE REVISIONS AS STANDARD

Last year a number of revisions of the Standard Methods of Fire Tests of Building Construction and Materials (E 119 – 41, formerly designated C 19 – 41) were published as tentative.[1] These included a closer definition of the time-temperature fire exposure curve and method of correcting indicated fire-endurance periods for variation of intensity of fire exposure from the standard curve, a new method of fire test of ceiling constructions, and an alternate method of conducting fire tests of structural steel columns without application of load. The latter is based on well-established relations between strength and temperature that enable the point of failure under load to be determined in terms of the attained temperatures of the steel. The committee, therefore, recommends that these tentative revisions, together with the following minor revisions, be approved for reference to letter ballot of the Society for adoption as standard:

Section 4.—Add the following clause to the last sentence in this section: "if temperatures at such points will obviously be lower than at more representative locations."

Section 5.—At the end of the new Paragraph (*b*), after the definition for "L", add the following: "(54 Fahr.-hr. or 30 Cent.-hr. (3240 Fahr.-min. or 1800 Cent.-min.))."

Present Sections 12 and 20.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

During the fire endurance and fire and hose stream tests [the construction shall be loaded] *a superimposed load shall be applied to the construction* in a manner calculated to develop theoretically, as nearly as practicable, the working stresses contemplated by the design.

Present Sections 13, 15, 18, 21, 24 and new Section 29.—Change the first part to read as follows by the addition of the italicized words and the omission of those in brackets: "The test shall [not] be regarded as successful [unless] *if* the following conditions are met:"

Present Sections 13 (*b*) *and 21* (*b*).—From line 8 to the end of these paragraphs, change to read as follows by the addition of the italicized words and the omission of those in brackets: "shall sustain [a

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 293 (1946); also, 1946 Book of A.S.T.M. Standards, Part II.

total load equal to] the dead load *of the test construction* plus twice the superimposed load specified above."

New Section 28.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

28. The test ceiling construction shall include all [the] structural members and details including hangers, if any, but [shall] not [include] walkways. Above the ceiling during a test *there* shall be provided a tight [enclosing construction with flat roof placed with the lower edge of its supports not more than 30 in. above the ceiling joists.] *flat-topped enclosure, the underside of the covering material of which shall be 36 in. above the top of the joists or beams supporting and protected by the ceiling.* The top of the enclosure shall be made of cement-asbestos board $\frac{1}{4}$ in. in thickness under asbestos millboard $\frac{1}{2}$ in. in thickness and the side walls of 8-in. common brick, or it shall be of a construction having equivalent heat conductivity and heat capacity. Where the use of a ceiling under a combustible construction is contemplated, at least five [1-in. pine boards each having an area of 2 sq. ft. shall be attached to the top of the enclosure under which the exposed surface temperatures can be measured.] *15-in. square panels of 1-in. pine boards shall be attached to the underside of the top of the enclosure. The temperatures on the bottom surface of these panels shall be measured.*

New Section 29(b)(2).—Change the first sentence to read as follows by the addition of the italicized words: "With combustible supports or other combustible material not in contact with the ceiling, the temperature increase on the surface of any combustible members, *pine panels*, or combustible material adjacent to the ceiling shall not exceed 250 F."

II. ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Method of Test for Combustible Properties of Wood by the Fire-Tube Test Method (E 69 – 46 T) be approved for reference to letter ballot of the Society for adoption as standard with the following revision:

Section 3.—Add the following as a new Paragraph (*b*) and reletter the subsequent paragraphs accordingly: "(*b*) Not less than 5 tests shall be made from each sample, except where the quantity is less than 2000 bd. ft. the number of tests shall be left to the discretion of the testing authority but shall be not less than two."

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

GENERAL ACTIVITIES OF COMMITTEE

Fire Tests of Partially Impregnated Wood.—Further consideration has been given by Subcommittee II on Fire Tests of Lumber, (Captain H. K. Hughes, USNR, chairman), to methods of conducting fire tests of wood, mainly of 2-in. or greater thickness, which has been given impregnation treatment with fire-retarding chemicals that in significant amounts do not penetrate the full section. In general it is not practical to obtain depositions effective in decidedly reducing the flammability to greater depths than $\frac{3}{8}$ to $\frac{1}{2}$ in. from the face for most species. This, however, would represent a considerable gain in fire safety for exposed structural members that are treated after fabrication or otherwise not cut to expose untreated surfaces. Quite possibly the present methods of test for wood given full impregnation can be adapted for the purpose with suitable sampling requirements.

Fire Tests of Window Assemblies.—Subcommittee III on Fire Tests of Wall Opening Protectives, (A. L. Brown, chairman), in a report to the main committee, which was approved, recommended that work on proposed specifications for fire test of window assemblies be suspended pending development of further information on the need for such a standard and the feasibility of framing suitable performance requirements. Glass being subject to fracture and fusion as exposed to fire, doubt was expressed whether the allowable fire effects could be stipulated without being either too permissive or too restrictive. The test may also call for closer control of the fire exposure than for constructions less sensitive to variations in test conditions.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at A.S.T.M. Headquarters.

Fire Tests of Acoustical and Similar Finishes.—A considerable volume of new data has accrued from tests by several methods during the past year. Decided interest has been expressed in the standardization of a testing procedure according to which materials used for interior finish in buildings can be evaluated from the fire hazard standpoint and it is hoped this will be realized as the result of this activity.

Nomenclature and Definitions.—It has been appreciated for some time that responsibility rests with the committee to define the terms used in connection with its activities. Subcommittee V on Nomenclature and Definitions, (J. R. Shank, chairman) has made a new start on this project which should contribute to precision and uniformity in the use of terms relating to fire hazard and fire resistance.

This report has been submitted to letter ballot of the committee, which consists of 52 members; 34 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

S. H. INGBERG,
Chairman.

H. M. ROBINSON.
Secretary.

REPORT OF COMMITTEE E-6*
ON
METHODS OF TESTING BUILDING CONSTRUCTIONS

Committee E-6 on Methods of Testing Building Constructions held a well-attended meeting on June 25, 1946, at Buffalo, N. Y., during the Annual Meeting of the Society. The work of the subcommittees has not progressed sufficiently to necessitate a subsequent meeting of the main committee during the year.

Much consideration was given by the officers during the year to the completion of the organization of the committee, and particularly to the establishment of the four additional subcommittees which were authorized at that meeting, as follows:

Subcommittee:

V. On Durability (Weathering, Exposure and Moisture Penetration), D. E. Parsons, chairman,

VI. On Insulation, F. G. Hechler, chairman,

VII. On Sound Transmission, R. K. Cook, chairman, and

VIII. On Fire Resistance, Harold Perrine, chairman.

The tentative plan of procedure for each of these subcommittees, the work of which must integrate with existing A.S.T.M. committees in the respective fields, contemplates a review of current methods and practices to determine whether they meet the anticipated needs which may arise in the evaluation of various building constructions in Committee E-6. If a subcommittee concludes from its review that standard methods of test are already available to meet the requirements of Committee E-6 in that particular field, then appropriate reference to such procedure can be made by the committee in standards and specifications which it may develop. However, if a subcommittee finds deficiencies in present adopted standards or gaps that need to be filled in, covering, for example, tests of constructions rather than materials, it will devolve on the subcommittee to see that appropriate standards are developed. Where such additional standard methods may be necessary, it is expected that these will be developed by or with the cooperation of existing A.S.T.M. committees in these fields.

Recommendation Accepted by the Administrative Committee on Standards

Subsequent to the 1946 Annual Meeting, Committee E-6 recommended to the Society through the Administrative Committee on Standards that Methods of Conducting Strength Test of Panels for Building Constructions be published as tentative. This recommendation was accepted by the Standards Committee on May 7, 1947, and the new tentative will appear in the 1947 Supplement to Book of A.S.T.M. Standards, bearing the designation E 72–47 T.

Activities of Subcommittees

Subcommittee I on Panels for Light-Building Construction (H. L. Whittemore, chairman) prepared the Tentative Methods E 72 which describe in detail methods for determining the strength and behavior of building constructions under compressive, transverse, impact, concentrated, and racking loads; also, contain provisions for reporting the test results. Although accepted unanimously by the committee, the subcommittee recognizes that there are several matters to which further consideration must be given in these methods. Experience with the use of the tentative test methods and further consideration by materials engineers should provide a basis for any needed improvement and modification.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

It is contemplated that these structural test methods will provide a means of obtaining comparable strength data on many constructions now coming into use for small buildings, and will thus provide a basis for evaluating the acceptability from this standpoint.

Subcommittee II on Connections and Assembled Structures (G. M. Rapp, chairman).—The work of this subcommittee is closely related to that of Subcommittee I, and the two subcommittees have collaborated in the formulation of the Tentative Methods E 72. As a result, a separate program has not yet been charted for Subcommittee II. However, a method of testing a specific type of building assembly comprising prefabricated welded steel roof trusses is being prepared for submission to the subcommittee.

Subcommittee III on Large Structural Units (F. E. Richart, chairman) has been engaged in the preparation of a preliminary draft of a method of test for roof trusses. A number of test methods were examined and a draft of the proposed method has been written and distributed to the subcommittee for criticism. It is hoped that this method of test, with any revisions resulting from comments from the subcommittee, will be completed for submission to Committee E-6 at its next meeting, and that it can be submitted to the Society as tentative during the coming year.

Subcommittee IV on General Structural Elements (K. F. Wendt, chairman) is engaged in the preparation of a proposed method of test covering expanded, welded, and rolled light-steel joists, precast reinforced concrete floor and roof slabs, and assembled floor and roof slabs such as clay tile or concrete block and reinforcing steel. While this work is progressing slowly the subcommittee is substantially of one mind on the following points:

(*1*) A single method of test applicable to all constructions should be developed rather than a series of methods for each special type.

(*2*) The method of test should follow as closely as possible the form suggested for a method applicable to ultimate consumer goods as shown in the October, 1946, ASTM BULLETIN[2] and should adopt whenever possible the apparatus and methods of test described in BMS2[3] as modified by Committee E-6.

(*3*) Panel tests should be prescribed for joist-type constructions which require lateral support or which depend on a floor or deck for support.

Many details of a standard procedure also have general approval of the subcommittee. A report for consideration by the main committee should be available early in 1948.

Subcommittees V, VI, VII, and VIII have been organized so recently that there has been no opportunity for meetings or consideration of their programs. However, it is expected that meetings of these subcommittees will be arranged during the Annual Meeting in June.

This report has been submitted to letter ballot of the committee which consists of 44 members; 40 members returned their ballots, a 11 of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. J. MARKWARDT,
Chairman.

J. H. COURTNEY,
Secretary.

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[2] Suggested Test Method Form Applicable to Ultimate Consumer Goods, A.S.T.M. BULLETIN, No. 142, October, 1946, p. 44.

[3] Herbert L. Whittemore and Ambrose H. Stang, "Methods of Determining the Structural Properties of Low-Cost House Constructions," *Building Materials and Structures, Report BMS2*, Nat. Bureau Standards, August 10, 1938.

REPORT OF COMMITTEE E-7*
ON
RADIOGRAPHIC TESTING

Committee E-7 on Radiographic Testing met at Atlantic City, N. J., on June 16, 1947, in conjunction with the Annual Meeting of the Society. Previous to this meeting the committee met in conjunction with the annual meeting of the Society in June of each year from 1941 to 1946. Brief progress reports of the committee activities were presented but not included in the Proceedings.

A symposium was conducted in Cleveland, Ohio, in February, 1946, on "Ultra High Voltage and High Speed Radiography." Eight papers were presented as follows:

"The Betatron," by Dr. D. W. Kerst.

"Discussion of Naval Research Laboratory Betatron," by Dr. H. F. Kaiser.

"European Induction Accelerators," by Dr. H. F. Kaiser.

"Electron Accelerator and the Two Million Volt Resonance Transformer Radiographic Equipment," by Dr. E. E. Charlton.

"Radiography with the Two Million Volt Electrostatic Generator X-Ray Machine," by Dr. R. J. Van de Graaff and Associates.

"Discussion of the Practical Application of the Van de Graaff Electrostatic X-Ray Generator,"[1] by D. T. O'Connor.

"One Millionth Second Radiography and Its Development,"[2] by C. M. Slack and L. F. Ehrke.

"Frankford Arsenal Experience with High Speed Radiography,"[2] by E. R. Thilo.

It was intended to make these papers available, together with the discussion, in a volume for distribution but the publication has suffered delays due to apparently unavoidable causes and is still under way. Another symposium is planned for 1948, and the papers from this symposium will be added to the 1946 papers for publishing in one volume next year.

The Executive Council held a meeting in October, 1946, during which one special subcommittee was discharged and two new subcommittees were appointed.

At the June, 1940, meeting of Committee E-7 a special subcommittee under the leadership of J. J. Curran was appointed to work on a "Procedures Manual for Radiographic Testing of Metal Castings." This manual was intended as a substitute during the war period for the Tentative Methods of Radiographic Testing of Metal Castings (E 15 - 39 T), which was known to be obsolete. Largely because the men who were to prepare the manual were busy with other war assignments, the project was never completed. It was decided at the Executive Council Meeting in 1946 to discontinue the work since it was no longer urgent. Accordingly, the special subcommittee was discharged with thanks for its efforts.

A new Subcommittee on Terminology under the leadership of K. R. Van Horn was appointed by the Executive Council. This subcommittee was charged with the responsibility for revision of the Tentative Radiographic Terminology for Use in Radiographic Inspection of Castings and Weldments (E 52 - 45 T), to meet criticisms received and to further perfect the terminology.

The Executive Council also appointed a new Subcommittee on Correlation of

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] ASTM Bulletin, No. 148, October, 1947, p. 57.

[2] ASTM Bulletin, No. 149, December, 1947.

Radiography with other Non-Destructive Tests with T. D. O'Connor as chairman to correlate radiographic testing with other non-destructive tests. While Committee E-7 is primarily concerned with radiography, the efficient use of this method requires knowledge of other non-destructive test methods. It is poor economy to use this method in places where others would work better or cheaper, and it is desirable, also, to know where radiography can be used as a valuable supplement to other methods of non-destructive testing. However, perhaps the most important question involved in correlation is an evaluation of radiographic results in terms of quantities derived from other tests, wheth r they be destructive or non-destructive.

In February, 1947, the Administrative Committee on Standards accepted the Tentative Industrial Radiographic Standards for Steel Castings (E 71 - 47 T). This action represents an adoption by the A.S.T.M. of a Navy Standard that was extensively used and tested during the war. The Navy Department has been most cooperative in that it has granted permission for reproduction of the radiographic negatives and has expressed its willingness to further assist the committee.

At the meeting of Committee E-7 on June 16, 1947, it was decided to recommend to the Society that the Tentative Methods of Radiographic Testing of Metal Castings (E 15 - 39 T) be withdrawn. A letter ballot on the question has been circulated to the members of Committee E-7. At this meeting some revisions in the by-laws of Committee E-7 were proposed to make them conform to present practice of the Society.

A Special Subcommittee on Standards for Weldments, O. R. Carpenter, chairman, was instructed to review the Navy Steel Welding Standards and prepare them in suitable form for presentation to the committee.

A Special Subcommittee on Standards for Light-Alloy Castings, K. R. Van Horn, chairman, was instructed to make recommendations concerning the feasibility of preparing radiographic standards for light-alloy castings for adoption as procurement guides rather than inspection standards. It was the consensus of opinion that standards for the latter purpose were not immediately feasible. This subcommittee was also charged with the responsibility for preparing a preliminary set of standards.

In view of the ever increasing field of non-destructive testing, other than radiography, which does not seem to be properly covered by the Society and in which the membership of Committee E-7 is greatly interested it was decided to request the Board of Directors that Committee E-7 be authorized to expand its scope. This will involve changing the name of the committee. The scope would be expanded to include all non-destructive testing for discontinuities in engineering materials. This matter is also to be submitted to letter ballot of Committee E-7.

Subcommittee II submitted to the committee a review of papers published between June 1, 1945, and June 2, 1946, including only those dealing with new information or developments in the physical or photographic elements.

Respectfully submitted on behalf of the committee,

H. H. LESTER,
Chairman.

J. H. BLY,
Secretary.

REPORT OF COMMITTEE E-9* ON FATIGUE

Since this is the first annual report of Committee E-9 on Fatigue, it is in order to record the basic facts concerning organization of the committee.

Committee E-9 was formed as a direct result of a recommendation by the Administrative Committee on Simulated Service Testing. The following is quoted from its 1946 Annual Report[1]:

> "The committee has recommended the reorganization of fatigue testing activities in the Society by establishing a new Committee E-9 on Fatigue, suggesting the organization, personnel, scope, etc. The scope has been assigned as follows:
>
> > To formulate methods for the determination of fatigue characteristics of simple and composite materials, components, and processed parts; to promote research in these fields; and to coordinate such Society activities conducted by other technical committees.
>
> The present Research Committee on Fatigue of Metals will be discontinued as such and its activities transferred to the new committee as a subcommittee on research.
>
> These arrangements have been approved by the Executive Committee and the new committee will be organized during the 1946 Annual Meeting."

An Advisory Committee was set up consisting of R. E. Peterson, chairman, M. A. Grossman, O. J. Horger, H. F. Moore, J. M. Lessells, and R. L. Templin. This committee laid much of the ground work for future activities of Committee E-9. Formal organization and election of officers of Committee E-9 occurred at Buffalo, N. Y., on June 24, 1946. Of the 53 committee members, 29 are from industry, 11 are from Government laboratories, 10 are from universities, and 3 are from independent laboratories.

Committee E-9 has initiated and is carrying forward the following three specific tasks:

Preparation of a Manual on Fatigue Testing: Considerable divergence now exists with regard to nomenclature, specimen design, methods of finishing specimens, methods of plotting data and interpretation of data. It is generally agreed that an authoritative manual will constitute a useful service on the part of the Society. First draft manuscripts are at hand for six of the eight sections.

Preparation of a List of Current Projects: There has been insufficient information concerning fatigue testing projects in progress, resulting in some duplication and inadequate cooperation. The list being prepared by Committee E-9 at present contains the programs of fourteen laboratories. In one case the list resulted in reorientation of a Government sponsored project.

Preparation of a List of Fatigue Subjects on Which Information Is Needed: Correspondence from universities and Government agencies indicates that it would be valuable to them in connection with theses and special research programs to know what Committee E-9 considers to be important questions needing attention. The list to date consists of fifteen suggestions.

In addition to these specific tasks, the usual functions of sponsoring papers, coordinating activities, etc., have been carried on. Twenty papers on fatigue have been reviewed by Committee E-9 resulting in four technical sessions and contributions to two joint sessions.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 95 (1946).

Committee E-9 has served as a clearing house for questions within its scope. In response to an inquiry from the Ontario Research Foundation, a circular letter was sent to all members of Committee E-9. The thirteen replies received were forwarded and have been helpful.

On two occasions the committee has assisted visiting European scientists by suggesting names of laboratory heads with whom arrangements for observing fatigue of metals research might be made.

The Committee E-9 organization which now exists makes it possible for the Research Subcommittee to devote its full time to discussion on a scientific level and two such sessions conducted under Professor Moore's leadership have proved to be most interesting and stimulating.

This report has been submitted to letter ballot of the main committee which consists of 43 members; 35 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. E. PETERSON,
Chairman.

O. J. HORGER,
Secretary.

REPORT OF COMMITTEE E-11*

ON

QUALITY CONTROL OF MATERIALS

Committee E-11 on Quality Control of Materials held its organization meeting in Philadelphia, Pa., on June 10, 1946. At this meeting the organization of the committee was completed. Permanent officers were elected, with H. F. Dodge as chairman, and a set of By-laws to govern the work of the committee was approved. A second meeting was held on February 25, 1947, in Philadelphia, Pa. Four meetings of the Advisory Committee have been held since its inception.

It is the plan of the committee in order to promote the knowledge of quality control methods and their application to specifications and methods of test, the purposes of its organization, that a series of manuals should be developed outlining procedures and practices in the field of quality control. The committee is now engaged in surveying the projects which should be undertaken, planning the work to be done, organizing task groups to carry on the work, and making the completed work available to the Society.

ORGANIZATION OF TASK GROUPS

At the present time four task groups have been organized. These groups and the scope of their work is as follows:

Group 1 on Presentation of Data (R. F. Passano, chairman) is preparing Sections 1 and 2 of the new proposed A.S.T.M. Manual on Quality Control of Materials, such sections to comprise editorial revisions of (*1*) main section of the A.S, T.M. Manual on Presentation of Data and (*2*) Supplement A thereof; the content is to be substantially unchanged, except that consideration is to be given to the omission of text material on skewness.

Group 2 on Control Chart Method of Analysis and Presentation of Data (A. E. R. Westman, chairman) is preparing Section 3 of the new proposed A.S.T.M. Manual on Quality Control of Materials, such section to comprise editorial revision of Supplement B of A.S.T.M. Manual on Presentation of Data; the content is to be substantially unchanged, except for modifications to treat separately charts for number of defects and charts for number of defectives, as covered in "Note Regarding April 1943 Reprinting," and to include additional examples, if needed.

Group 3 on Numerical Requirements in Standards (K. L. Fetters, chairman) is preparing a recommended practice based on the subject matter of Sections 3, 4, 5, and 6 of the Tentative Recommended Practices for Designation of Numerical Requirements in Standards (E 29 – 40 T) in a simplified form to be of maximum usefulness to specification-writing committees; and to make recommendations regarding ways and means of making available the remainder of material in Tentative Recommended Practices E 29 (either present content only or combined with additional material) in a form most useful to A.S.T.M. membership.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

Group 4 on Survey of A.S.T.M. Sampling Plans (G. H. Harnden, chairman) is making a survey of sampling plans used in A.S.T.M. standards and tentatives to disclose the variety and types of materials in such manner as to make statistical evaluation of these plans possible.

Organization of other task groups is under active consideration. It is expected that such subjects as sampling plans and planning for the collection of standardization data will be covered.

Members of Committee E-11 have attended meetings of other committees of the Society, at the request of the latter, and have discussed with them the use of quality control methods in the development of their standards.

This report has been submitted to letter ballot of the committee which consists of 21 members; 17 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. F. DODGE,
Chairman.

O. P. BECKWITH,
Secretary.

TECHNICAL PAPERS

With Discussions

EDGAR MARBURG LECTURE

The purpose of the Edgar Marburg Lecture is to have described at the Annual Meetings of the American Society for Testing Materials, by leaders in their respective fields, outstanding developments in the promotion of knowledge of engineering materials. Established as a means of emphasizing the importance of promoting knowledge of materials, the Lecture honors and perpetuates the memory of Edgar Marburg, first Secretary of the Society.

ENGINEERING LAMINATES
FUNDAMENTALS UNDERLYING THE PROBLEMS OF THEIR INHOMOGENEITY[1]

TWENTY-FIRST EDGAR MARBURG LECTURE

BY WALTER C. VOSS[2]

> "Science is always wrong. It never solves a problem without creating ten more."
>
> —*Bernard Shaw*

INTRODUCTION

When I was asked to prepare this paper to be delivered as the Edgar Marburg Lecture at this session of the American Society for Testing Materials, I was not aware of the many problems which I would have to face, and was lured into a position in which I would tread "where angels fear to tread." Inasmuch as engineering laminates cover a wide variety of natural and synthetic materials, and introduce the inhomogeneity of laminar assemblages and adhesives, it may be that some of our present methods of testing and of determining specific characteristics are inaccurate and misleading. I am well aware of the challenge which a discussion of this subject poses and which confronts the entire engineering profession. Therefore, having accepted the challenge of this task, it was necessary for me to approach the discussion by an examination of some of the basic science involved.

It will be well to review the steps through which we have arrived at some of our present knowledge of materials. The initiation of most research has been at the behest of some group which needed to know whether a material or assemblage of materials would accomplish a definite purpose for which there was a demand. Such a decision usually results in a program of applied research based upon the assumptions of certain variables which could be set at the time.

A great deal of the structure of such a plan may well be based upon the opinion of one or more people who may be unmindful of many pertinent variables which later on may prove to be troublesome. Many researches which have consumed much time but, nevertheless, have contributed immeasurably to a general over-all knowledge of the particular problem in hand, have been started in this way. Perhaps none of them has been completed without impressing upon the investigators the desirability of knowing why certain strange results were obtained. The inconsistency of results has often led to the admission that some basic fundamentals which controlled the problem were unknown. If these fundamentals could be postulated clearly, it might be possible to guide the search for them in a more direct way. We might then entertain the hope that a partial solution of the "why" of the problem would be brought to light. The experiments can then be repeated with more promise of success. For these reasons most research is a mixture of fundamental and applied research.

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[1] Read on June 18, 1947, before the Fiftieth Annual Meeting of the American Society for Testing Materials, Atlantic City, N. J.

[2] Head of Department of Building Engineering and Construction, Massachusetts Institute of Technology, and Consultant in Architectural Construction and Materials, Cambridge, Mass.

The classic approach to research involves certain very well-defined steps. These steps are: (*a*) survey of existing directly and indirectly related information, (*b*) correlation and study of such data for the purpose of formulating certain hypotheses, (*c*) the setting up of definite hypotheses, (*d*) testing these hypotheses, (*e*) formulation of a theory for continued study, and (*f*) the annunciation of a law after substantial trial and discussion. With these steps in mind the present status of our work with engineering laminates presents a curious and extremely interesting conglomerate of the first three steps in this approach. There are many investigators in the industry who are testing many combinations by more or less cut-and-try methods. They are thus contributing to the mass of information which must be studied thoroughly before anything of real consequence may emerge. This is a healthy situation and it is hoped that work of this kind will continue not only in amount but in quality of direction and detail. At the same time, it is necessary that those who are studying these results bring to bear upon their study some of the fundamental principles of science which are involved. The tendency to compartmentalization of our various sciences, which so besets us at the present time, has made it a bit difficult for those who possess the knowledge of the basic matters to weigh the requirements for particular applications. On the other hand, those who are well qualified to define what a material must do are often unable to understand the basic scientific aspects, and are perhaps unable to present to the pure scientist exactly the approach which they hope to have made. However, with the information from such surveys, made here and there, with or without a full conception of their import, it often becomes possible to evolve a more basic concept.

Fully cognizant of the history of our study of materials, I should like to ask you to follow with me some of the concepts which lie at the base of an appreciation of engineering laminates and which I had to explore before my thoughts on this matter assumed any consistent pattern. This will involve a discussion of some of the aspects of the theory involved in the structure of the atom and the bonds which exist; a consideration of crystallography as it applies to crystal structures, the lattice and the energies involved with their relation to this problem; and a rather simple study of the elusive subject of adhesives and their relation to surface energy and molecular forces. These form a necessary base for any discussion of engineering laminates as exemplified by the sandwich types of materials which we presently are using commercially. Much of this fundamental theory applies equally to many of our other engineering materials.

ATOMIC STRUCTURE AND BONDS

Appreciation of the fact that interatomic forces were responsible for the coherent stability of molecules dates as far back as the earliest of our modern chemistry. Over two hundred years ago Desaguliers (1742) suggested the electrical character of these forces.

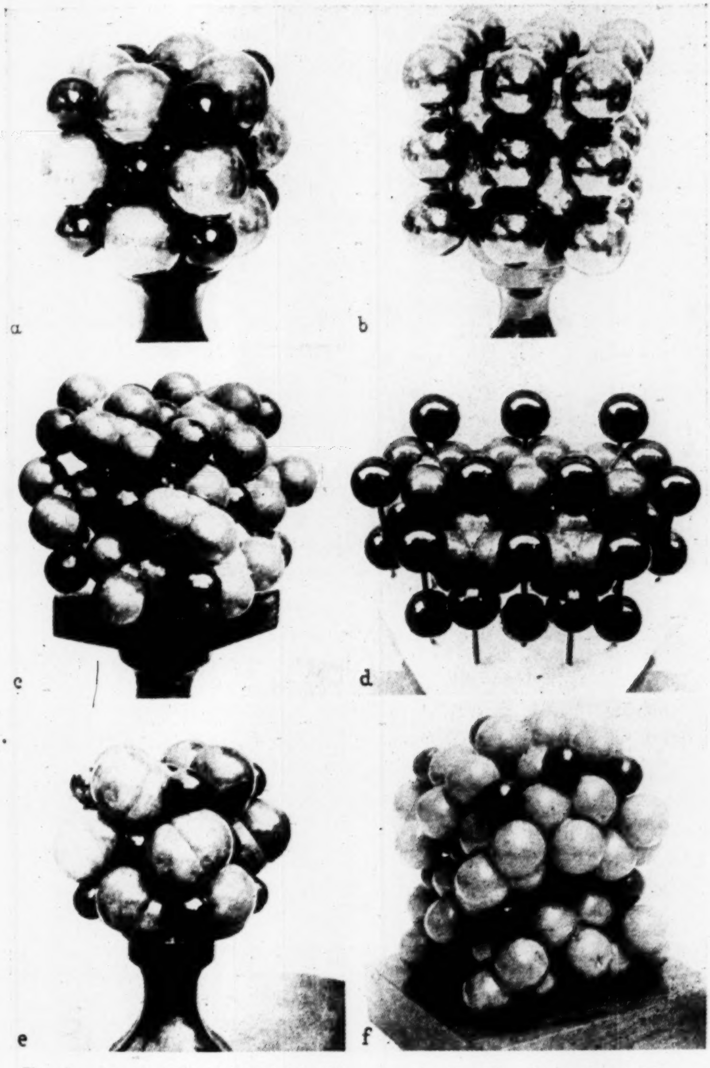


FIG. 1.—Crystal Models Which Illustrate the Packing Together of Ions.
Taken from "The Crystalline State," by W. H. and W. L. Bragg, G. Bell & Sons, Ltd.

One hundred and twenty-five years ago Berzelius (1819) developed this idea systematically. Forty years ago Barlow and Pope (1906) conceived a crystal as an essentially geometrical entity which is formed by a definite packing of spherical atoms, each of which had a definite characteristic size determined by the state of equilibrium which depends upon the attractive and repulsive forces acting between the neighboring atoms or ions (Fig. 1). It has been suggested by some of these early investigators and scientists that there is no essential distinction between the chemical forces that are responsible for binding the atoms together into the chemical molecule and the physical forces which establish the coherence of the solid as a whole. They claim that in the great majority of simple compounds the molecule as such does not exist in the solid state. Some thirty years ago Lewis and Kossell (1916), working separately, suggested that two types of binding forces account for the formation on the one hand of molecules such as NaCl containing atoms of essentially different character and on the other hand molecules such as $(Cl)_2$ composed of similar or identical atoms. It was also found at this time that the atom may occur in different bodies in different states of ionization and that it may have correspondingly different characteristic radii. Thus, the radius of the sodium atom in metallic sodium is 1.86 Å, but the radius of the sodium ion in sodium halides is 0.98 Å. Thus, physical as well as chemical properties have to be explained in terms of bonds, and because of this, a thorough knowledge of physical as well as chemical properties is necessary to throw enough light on the nature of these bonds. With the development of the modern quantum theory the problems of interatomic bonds have been further explored and have disclosed much valuable information on the nature of the forces involved. It is quite probable that we will have to look to the further development of quantum mechanics for our future advances in the interpretation of chemical forces. At present this theory can treat only with the very simplest of structure types.

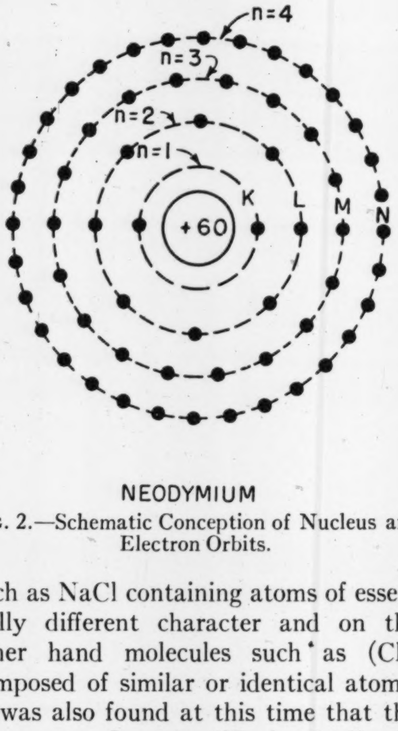


FIG. 2.—Schematic Conception of Nucleus and Electron Orbits.

Let us look at atomic structure in a very general and perhaps superficial way. Bohr's conception of the atom suggested that the nucleus had a diameter of 10^{-13} cm. and contributed practically the whole mass of the atom, while the atom as a whole had a diameter of 10^{-8} cm. or more. The nucleus was conceived as carrying a positive charge, relative to the charge on the negative electron, and this is considered equal to some integral number of times the unit electronic charge. This latter is called the atomic number. Surrounding this nucleus and making the atom as a whole electrically neutral is a set of electrons arranged in a series of orbits. This conception is shown in Fig. 2. The way in which the electrons are distributed over these orbits determines the chemical properties of the atom and governs the position of the element in the periodic classification. The overall mass of an atom is 1.65×10^{-24} g. $\times$ its atomic weight. The mass of an electron is 9.11×10^{-28} g. The charge on the electron is 4.80×10^{-10} electrostatic units, where the repulsion between two similar unit charges placed 1 cm. apart is equal to 1 dyne. It can thus be seen that two groups of electrons each weighing 1 g., if placed 1 cm. apart, will repel each other with a force of 5×10^{23} tons —a substantial force. The unit charges and masses of the fundamental particles comprising the atom are given in Table I.

The implication of these matters can be illustrated by an example using the helium nucleus. This nucleus contains two protons and two neutrons. Together these have a mass of 4.034 atomic weight units. However, the helium atom has a mass of only 4.003 atomic weight units. If we could construct a helium nucleus from separate protons and neutrons we would lose a mass of 0.031 atomic weight units. Albert Einstein made the bold assertion, "when mass apparently vanishes in a nuclear transformation, it is converted into energy." Assuming this to be true, the energy loss is $9 \times 10^{20} \times$ mass expressed in grams, where 9×10^{20} is the square of the velocity of light. Thus, 4 g. of helium produced from protons and neutrons would produce $9 \times 10^{20} \times 0.031$ ergs, or enough energy to provide electricity for a thousand average homes for a year.

TABLE I.—FUNDAMENTAL PARTICLES.

	Charge	Mass, atomic weight units
Electron	−1	0.00055
Positron	+1	0.00055
Proton	+1	1.008
Neutron	0	1.009
+Meson	+1	0.1 (approx.)
−Meson	−1	0.1 (approx.)

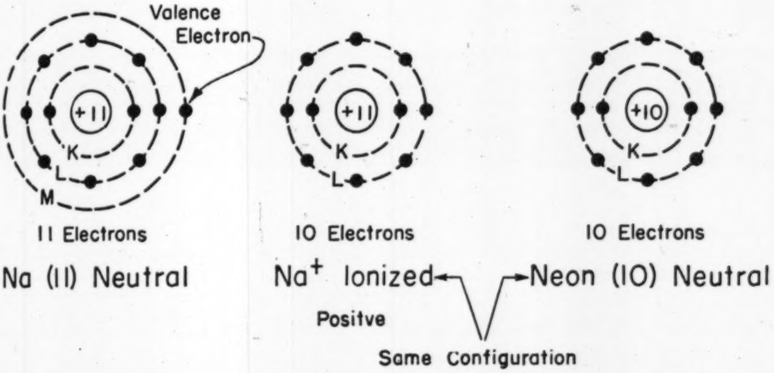


FIG. 3.—The Electronic Configuration of the Sodium Atom.

It is not suggested that the electrons in the various shells surrounding the nuclei of atoms are all in the same plane, but rather that they are in the trace of the electron orbit which may be circular or elliptical, and distributed according to the conditions which exist at the time. Figure 2 is used with this in mind for the purpose of clarity to show the relations between quantum numbers and the various extranuclear orbits lettered from *K* to *N*. The electrons with the total quantum number, $n = 1$, are in the *K* orbit, those with the quantum number, $n = 2$, are in an *L* orbit, $n = 3$, in an *M* orbit, and, $n = 4$, in an *N* orbit. It must be remembered that there are other quantum numbers which govern the shapes of the individual orbits. The orbital quantum number, l, expresses the ellipticity of the orbit and can have any integral value from n to $n - 1$. It must also be remembered that in general the lower the quantum number the greater the binding energy, which is the same as saying that the electrons in the *K* orbit have a higher ionization potential than those in an *L*, *M*, or other orbits. For the total quantum number 3, if $l = 0$, the orbit is circular; when $l = 1$ it is elliptical; when $l = 2$ the orbit is more elliptical. In other words, the ratio of the major to the minor axis becomes larger as l increases. The number of electrons necessary to complete an orbit with quantum number n is $2n^2$ and the number required to complete a subgroup of quantum number l is $2(2l + 1)$. Aside from the complications introduced by some of the rare earth elements and some of the still unknown elements in the periodic table, we find that the *K* orbits may have a maximum of two electrons; the *L* orbits a maximum of eight electrons; the *M* orbits a maximum of eighteen electrons; and the *N* orbits a maximum of thirty-two electrons.

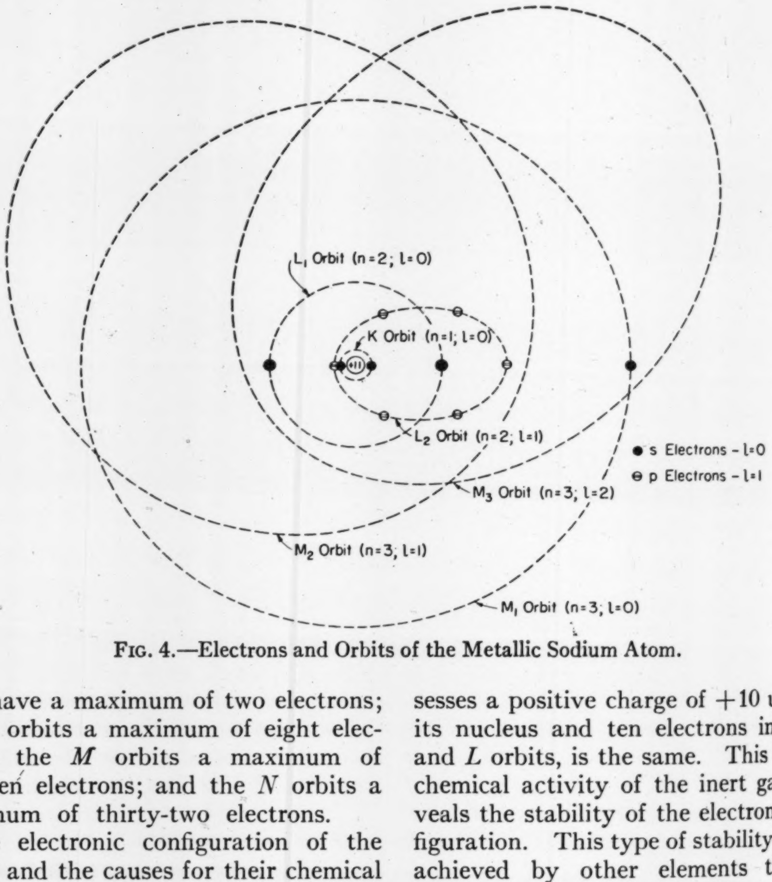


FIG. 4.—Electrons and Orbits of the Metallic Sodium Atom.

The electronic configuration of the atoms and the causes for their chemical affinity with other atoms as well as their relation to some of the neutral gases is illustrated in Fig. 3 for the sodium atom. It will be noted that neutral sodium (Na), which has a single valence electron in an *M* orbit, and has a total of eleven electrons neutralizing the charge in the nucleus, may be transformed by ionization, as shown, by the loss of the electron in an *M* orbit. This leaves only ten electrons. The charge in the nucleus remains the same. The ionized sodium (Na$^+$) therefore has a positive charge of 1 unit. It will also be noted that the configuration of the ionized sodium and the gas Neon, which possesses a positive charge of $+10$ units in its nucleus and ten electrons in its *K* and *L* orbits, is the same. This lack of chemical activity of the inert gases reveals the stability of the electronic configuration. This type of stability can be achieved by other elements through the loss or gain of one or more electrons. Figure 4 shows the metallic sodium atom in a different way. Because of the conditions which stimulate electronic activity, it is possible to have an *s* electron jump to a *p* orbit. As shown before, the *s* electrons are in the orbit which has an *l* value of 0 and therefore is circular, while the *p* electrons with an *l* value of 1 are in an elliptical orbit. Thus, in the *L* orbits we have two *s* electrons and six *p* electrons approaching the *M* orbits at certain points. Again it must be remembered that these are shown as planar electron values and are really distributed in three dimensions. This situation poses a very interesting hypothesis. Electrons rotating in the elliptical *L* orbits may at a particular moment be very near some adjacent atom, which because of the particular charge on the atom as a whole and perhaps because of the closeness of the electrons due to the conditions existing, may be very vigorously attracted forming what may be the essence of a very firm bond. If this hypothesis is correct, it also indicates that the production of bond by this method is one of circumstance and may not be of the type of continuity which effects the results wanted. However, if continuous bonds can be obtained over a surface, it will be possible to have sufficient adhesion to give all of the characteristics necessary for the particular use of the material. Scientifically it is necessary to predict the probability of sufficient continuity so the test of the bond would give exact and reproducible results.

It would appear, therefore, that many molecules even though neutral in themselves can still exert electrical forces because the charges are not symmetrically distributed over their apparent surfaces. Molecules in this state are called polar and their polarity is measured by their dipole moment. At higher temperatures, the molecules of liquids and gases rotate. In this state of motion there is very little opportunity for action between them and the surface ions of a crystallographic lattice. Any attractions or repulsions in this stage are impermanent. As the temperature falls, rotation changes to oscillation about positions of equilibrium induced in the particular area. The molecules can then orient themselves and bipolar attraction results. This is quite likely the beginning of a state of adhesion. Even nonpolar molecules can be oriented by the proximity of electrically charged particles, if polarizable, and thus induce a dipole bond.

Due to our lack of knowledge of the scientific bases for establishing the continuity of such bond, we must often disregard the results of our applied tests. We should be stimulated to explore the scientific bases for such inconsistencies and, if possible, find the sources of error. This may be a very complicated and complex thing to do. The only other alternative is to make sure that we allow a large factor of safety.

Because of the low compressibility of all ionic crystals, it is evident that the forces acting vary rapidly with the interatomic distance and much more rapidly than would be indicated by the inverse square law of Coulomb attraction. It would thus become a tenable hypothesis that we may regard the ions in an ionic structure as approaching rigid charged spheres of characteristic constant radius.

On the basis of our present knowledge of interatomic bonds we have recognized four types as follows: ionic, homopolar, metallic, and van der Waals. What we have discussed thus far has been related essentially to the ionic bond which is illustrated, for example, by the coherence of a substance such as NaCl. These materials form strong hard crystals. The homopolar bond, which also forms strong hard crystals, is illustrated by a structure such as the diamond. The metallic bond is illustrated by most of the pure metals, which possess variable strength and considerable malleability. This bond is responsible for the coherence of a metal. The van der Waals' bond, often called residual bond, is responsible for the coherence of inert gases when condensed to solids at low temperatures. When only one type of force occurs, the crystals are said to be homodesmic, and when two or more different types of bonds are in operation together the structure is termed heterodesmic. In the heterodesmic structure the physical properties such as hardness, mechanical strength, and melting point, are in general determined by the weakest bond present in the structure.

The strength of the ionic bond is revealed by the mechanical strength, hardness, and relatively high melting point of all the crystals. An increase in the size of the metallic ion brings about an increase in the *A-x*, or interatomic, distance and this results in a corresponding reduction in bond strength, immediately reflected in a rapid decrease in hardness. This is illustrated in Table II for the succession of compounds given.

It will be noted from Table II that as the interatomic distance increases the hardness decreases materially (*a*). The table, (*b*), also shows the result of substituting for the anion, one of greater radius. Thus, when calcium is in turn combined with sulfur, selenium, and tellurium, the hardness decreases but not at as great a rate as shown in (*a*). The bond strength is also affected by a change in the charge of the ion as shown in (*c*). In these instances where the interatomic distance is approximately the same, but the ionic charge increases, the hardness increases with the ionic charge. The melting points are affected by these changes in much the same way.

TABLE II.—HARDNESS AS AFFECTED BY ATOMIC STRUCTURE.

	BeO	MgO	CaO	SrO	BaO	
(*a*) *A-x* (interatomic) distance, Å	1.65	2.10	2.40	2.57	2.77	Effect of interatomic distance
Hardness, mohs	9.0	6.5	4.5	3.5	3.3	
	CaO	CaS		CaSe	CaTe	
(*b*) *A-x* distance, Å	2.40	2.84		2.96	3.17	Effect of anion radius
Hardness, mohs	4.5	4.0		3.2	2.9	
	NaF	MgO		SeN	TiC	
(*c*) *A-x* distance, Å	2.31	2.10		2.23	2.23	Effect of ionic charge
Hardness, mohs	3.2	6.5		7 to 8	8 to 9	

TABLE III.—THERMAL EXPANSION (α) AS AFFECTED BY ATOMIC STRUCTURE.

	NaF	NaCl	NaBr	NaI	
(*a*) *A-x* (interatomic) distance, Å	2.31	2.79	2.94	3.18	Effect of interatomic distance
$\alpha \times 10^6$	39	40	43	48	
	CaF_2				
(*b*) *A-x* distance, Å	2.39	Effect of divalence			
$\alpha \times 10^6$	19				

Thermal expansion is similarly affected as might be expected, the results for the sodium halides being shown in Table III.

It should be noted from Table III that the expansion coefficient of ionic halides of divalent metals, (*b*), are considerably smaller. Thus, CaF_2 with an interatomic distance of 2.39 has an expansion coefficient of 19×10^{-6}, as compared to NaF which has an expansion coefficient of 39×10^{-6} and essentially the same A-x distance.

For the homopolar bonds, of which diamond and zincblende are typical examples, we find the mechanical properties are similar in many respects to those for ionic bonds. Generally speaking, homopolar bonds are stronger than ionic bonds and may be effected by a single element such as diamond, where one carbon atom is surrounded by four others or by a combination of two elements as in zincblende, where one zinc atom is surrounded by four sulfur atoms.

The present general theory of metallic bond accepts Drude's idea that a metal is an assemblage of positive ions immersed in a gas of free electrons. He claimed that the attraction between the positive ions and the electrons was responsible for the structural coherence of metals and that the free mobility of this electron gas or cloud under the influence of electrical or thermal forces was responsible for high conductivity. His theory, however, gave us very little information regarding the nature of the interatomic binding forces in a metal. The forces responsible for the coherence of the metal are now considered to be chemical in nature, and are related to the chemical valence theory in some very distinct way. These two approaches to the understanding of atomic bonds have been reinforced by recent work on the theory of the metallic state.

Present thought on the bond in metal is that the bonds from one atom must be regarded as spherically distributed and to be capable of acting on as many neighbors as can be packed around that atom. The very highly coordinated structure of most of the true metals seems to bear out this theory. Even in the molten state this highly coordinated structure occurs. This seems to be substantiated by the small volume change found when metals move from the molten to the solid state. Metallic bond resembles the ionic in its order of coordination, but differs from the ionic bond in that it is exerted between two atoms not of chemically widely different character but between either identical atoms, as in the metallic elements, or between chemically similar atoms, as in an alloy system. In the metallic system there is no necessity for electrical neutrality prescribing the number of atoms of each kind which should occur, and the structure which results is determined almost solely by geometrical consideration. It would thus seem that the interchanges are very flexible and extensive and thus would confuse many who try to interpret alloy systems in terms of chemical combinations based upon properties of ionic and homopolar bonds.

The physical properties of most of the pure metals can be explained in terms of the mobility of the free electrons. Thus, the thermal conductivity and expansion, electrical conductivity, optical opacity or high reflectivity are all affected by this phenomenon. Although the electrons are able to move readily throughout the structure and are held within the solid as a whole by potential fields at the surface of the solid, electrons cannot be removed from the solid unless the work function corresponding to the minimum amount of energy which must be imparted to the electrons exceeds the height of the surface potential barrier. Metals may therefore be regarded as bound in a crystal orbital which embraces the entire structure. The great malleability of metals distinguishes them from most other solids. The undirected nature of the metal bonds and the very simple structure give rise to this property, and as a result crystals may glide upon each other by a relatively small displacement of closely packed layers in the lattice. When foreign atoms disturb the regularity of this structure, the metals become more brittle or at least less malleable.

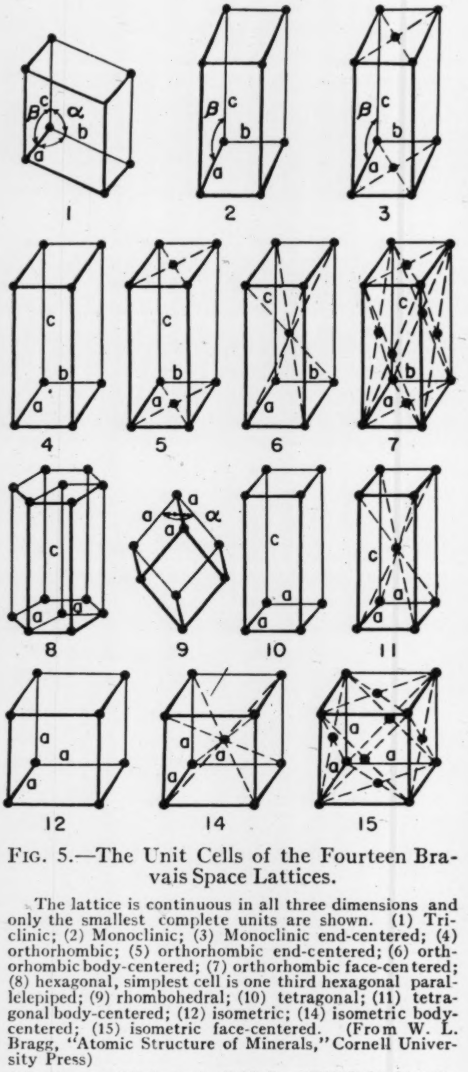


FIG. 5.—The Unit Cells of the Fourteen Bravais Space Lattices.

The lattice is continuous in all three dimensions and only the smallest complete units are shown. (1) Triclinic; (2) Monoclinic; (3) Monoclinic end-centered; (4) orthorhombic; (5) orthorhombic end-centered; (6) orthorhombic body-centered; (7) orthorhombic face-centered; (8) hexagonal, simplest cell is one third hexagonal parallelepiped; (9) rhombohedral; (10) tetragonal; (11) tetragonal body-centered; (12) isometric; (14) isometric body-centered; (15) isometric face-centered. (From W. L. Bragg, "Atomic Structure of Minerals," Cornell University Press)

After Norton, "Refractories," McGraw-Hill Publishing Co.

Although van der Waals' bond was supposed to apply to liquids and gases, it is now believed that it applies to some extent in solids. Debye (1920) described this bond as the forces which are acting as a result of the polarization of the electronic configuration of each atom by the field of its neighbors. This polarization resulted in the attraction between the dipoles thus created. The van der Waals' bond is comparatively weak and thus would indicate high compressibility, low melting point and high thermal expansion. The forces which hold the chain crystals of the type of cellulose, rubber and fibrous protein together in groups which are of the heterodesmic type, result from the action of van der Waals' bond. This bond being weak, as stated above, results in forces between molecules which are much weaker than those inside the molecule.

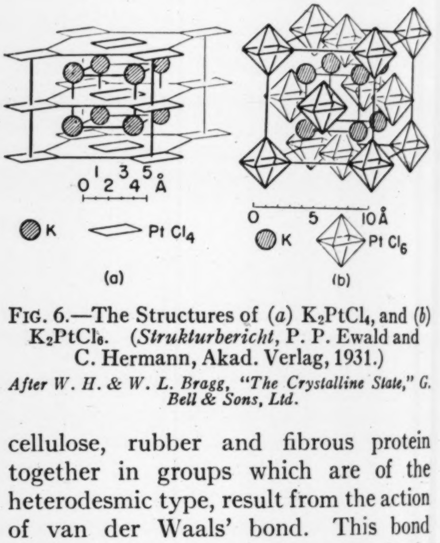


FIG. 6.—The Structures of (a) K_2PtCl_4, and (b) K_2PtCl_6. (*Strukturbericht*, P. P. Ewald and C. Hermann, Akad. Verlag, 1931.)

After W. H. & W. L. Bragg, "The Crystalline State," G. Bell & Sons, Ltd.

CRYSTAL LATTICES

Let us now consider the general theory of lattice structures which has been developed through the efforts of physicists and crystallographers. Among the most common lattices are the planar and space groups. The 14 Bravais space lattices are shown in Fig. 5. Those space groups which are based upon rotors or axes repeat a motive two, three, four, or six times. While a discussion of crystallography in some of its more basic aspects would be interesting and valuable in solving some of our problems in connection with engineering laminates, it is my intention to call only a few fundamental situations to your attention, which may help to clarify my approach and to stimulate further consideration of such matters in connection with these materials.

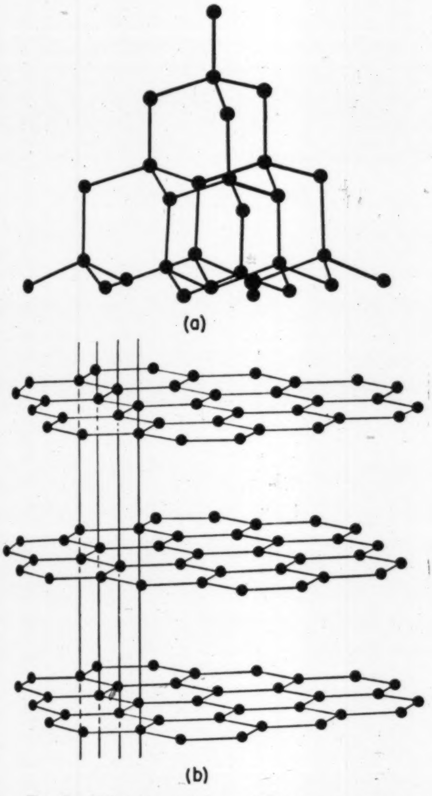
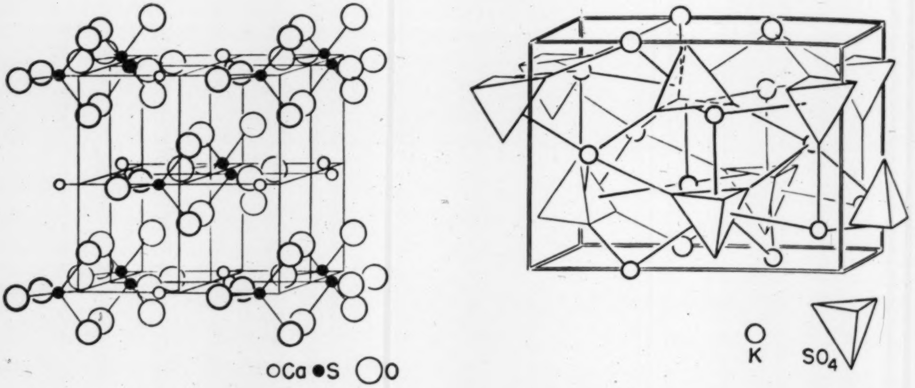


FIG. 7.—The Structures of Diamond and Graphite.
(*a*) Diamond
(*b*) Graphite
After W. H. and W. L. Bragg, "The Crystalline State," G. Bell & Sons, Ltd.

To illustrate the relations between the usual qualitative and quantitative chemical analyses and the present knowledge which we obtain from crystallography, a reference is made to Fig. 6. In this figure the structure of potassium chloroplatinate (K_2PtCl_4) and potassium hexachloroplatinate (K_2PtCl_6) are shown. It will be noted that K_2PtCl_4 is a plate structure held together with bonds to the potassium ions. When two additional ions of chlorine are added we get the octahedral type of structure held together with potassium ions. These two crystals behave quite differently as can readily be seen from the crystallographic structure. It would be indicative of the structure of (K_2PtCl_4) to provide easy gliding, whereas for the (K_2PtCl_6) this characteristic would of necessity disappear. While a quantitative chemical analysis for both of these compounds would be the same in most respects, X-ray diffraction crystallography indicates considerable difference. Likewise, we might consider the structure of two types of materials made of carbon. These two, the diamond shown in Fig. 7 (*a*) is a space lattice with substantial interatomic bond. The lattice of graphite indicates a series of planar homopolar bonds between the carbon atoms with little or no bond between the various layers. In connection with graphite we find that the distance between the neighbors in a single sheet of atoms is about 1.42 Å and that the spacing of the sheets is 3.4 Å. The bonding within each sheet is homopolar or co-valent, and the sheets are held together by the weaker van der Waals' forces. This checks very well with the softness of graphite and the easy sliding characteristics which impart lubricating qualities to this material. Even though a qualitative chemical analysis would show the same results for these two materials, the chemical analysis could not hope to show some of the characteristics which crystallographic methods have shown. Another illustration of the variations between lattice structures,



(a) $CaSO_4$ (gypsum)

(b) K_2SO_4

FIG. 8.—Space Lattices for $CaSO_4$ and K_2SO_4.

From R. C. Evans, "An Introduction to Crystal Chemistry," Cambridge Univ. Press

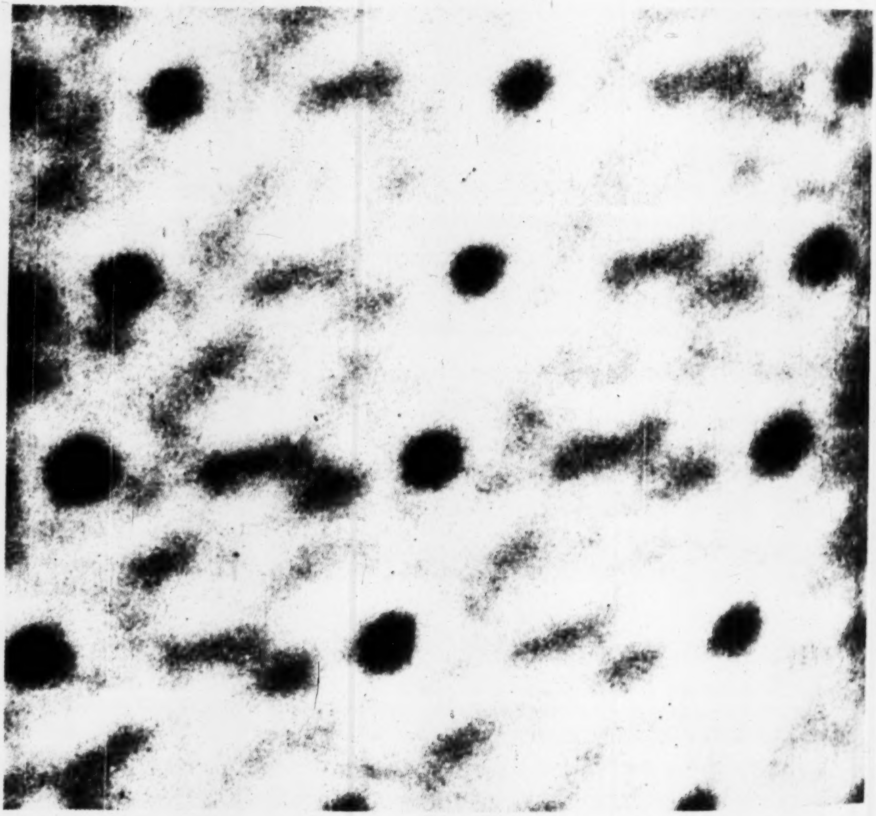


FIG. 9.—Image of Crystal Structure, Projected on Face (010). (*Zeit. f. Krist.*, Vol. 70, p. 489 (1929).)

After W. H. and W. L. Bragg, "The Crystalline State," G. Bell & Sons, Ltd.

because of the use or substitution of a different metal, is illustrated in Fig. 8 in which (*a*) gives the space lattice for $CaSO_4$ (gypsum) and the second (*b*) the space lattice for K_2SO_4.

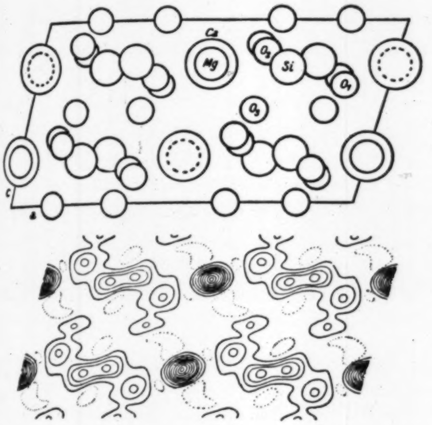


FIG. 10.—Projections of the Diopside Structure Upon (010). (*Zeit. f. Krist.*, Vol. 70 p. 488 (1929).)

After W. H. & W. L. Bragg, "The Crystalline State," G. Bell & Sons. Ltd.

A very interesting relationship exists between the space lattice as established by X-ray and crystallographic means, the actual X-ray image of the crystal structure and the contour lines of electron concentration as developed by Borne and Lande and later given accurate quantitative expression by Compton. Figure 9 shows the X-ray image of the crystal structure of diopside [$CaMg(SiO_3)_2$], a calcium magnesium metasilicate, projected on the 010 plane. Figure 10 shows the structure of diopside on the same plane together with the projection of the contours of electron concentration established by mathematical means.

Figure 11 shows the effect of heating sodium chloride, on the vibration of the atoms. It will be noted that as the salt is heated the number of electrons per Angstrom unit decreases and the spacing in Angstrom units is increased. This illustrates the tendency for expansion due to heat and in the case of this particular salt heat is lost in electron activity. It is not my intention to go into this phase of mathematical derivation and those who are further interested should consult such authorities as Evans and Bragg.

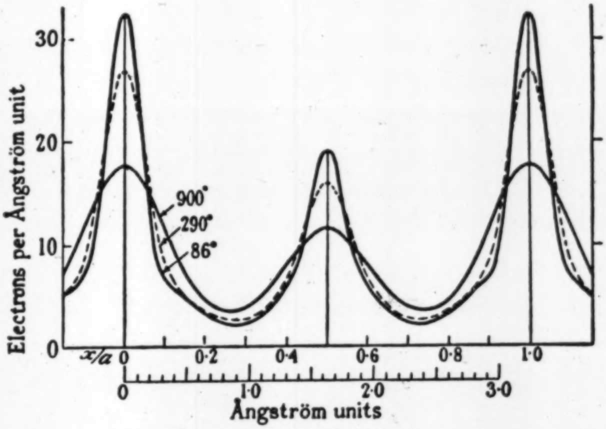
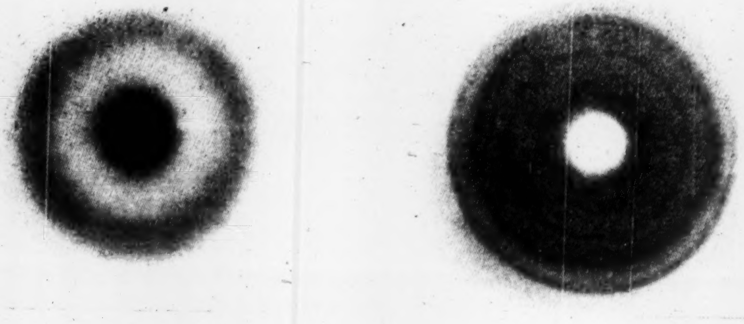


FIG. 11.—The Vibration of the Atoms When NaCl Is Heated. The octohedral crystal planes consist of alternate sheets of chlorine and sodium atoms, represented in the figure by the large and small peaks respectively As the temperature is raised the sheets become broader and more diffuse, owing to thermal movement (James and Firth). (*I.E.E., Journal*, Vol. 69, p. 1243 (1931).)

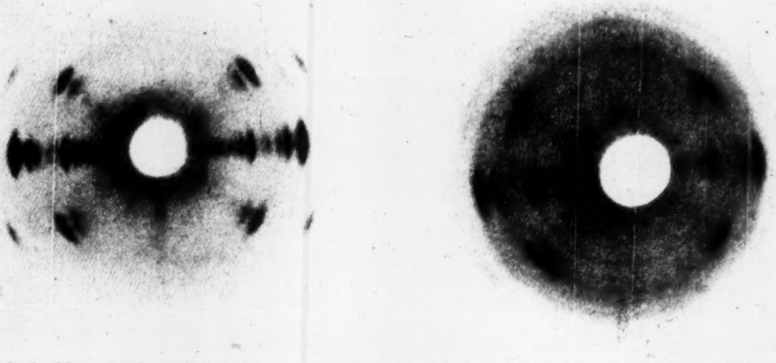
After W. H. & W. L. Bragg, "The Crystalline State," G. Bell & Sons, Ltd.

When lattices are put under stress they may be strained elastically, plastically, or both. Elastic deformation is usually temporary and not likely to cause rupture. Plastic deformation may be pure translation or twin gliding. In the first type the crystallographic elements remain oriented in planes parallel to the original position. Gliding occurs along atomic planes in very definite directions. This causes no change in the orientation of the crystal structure.



(*a*) Unstretched rubber, showing simple amorphous band.

(*b*) Unstretched frozen rubber showing Debye-Scherrer-Hull circles.



(*c*) Racked rubber, showing only interference spots (the amorphous band has entirely disappeared).

(*d*) Stretched frozen rubber, showing Debye-Scherrer-Hull circles with superposed interference spots.

FIG. 12.—X-ray Diagrams of Rubber.

From E. A. Hauser, "Colloid Chemistry of the Rubber Industry," Oxford Univ. Press

PLASTICS

The tremendous impetus that was given to our knowledge of plastics during the war was due in part to the fact that plastics themselves are rationally integrated compounds. They are produced from organic compounds such as coal, petroleum, acetylene, plant or animal products and contain carbon with hydrogen and oxygen or nitrogen and a very few other elements. They, therefore, are unable to withstand high temperatures without decomposition. The inorganic silicones, on the other hand, resist high temperatures. The organic plastics play an important rôle in the adhesives presently being used in engineering laminates and it will be wise for us to explore some of their characteristics in some detail.

For a long time many of the organic compounds were considered to be distinctly amorphous and lacking the structure of crystals. Today we know that they do possess organic crystalline structure, that the molecules may be long and narrow as in the long-chain compounds, flat as in aromatic compounds, or a globular cluster with complicated interconnections as in some of the thermosetting plastics. The binding forces may very well be of the van der Waals' type as in the hydrocarbons. These forces are so weak and undirected that the form of the crystal is almost entirely determined by the packing together of molecules of characteristic shape as for anthracene and paraffine. The molecules may have local dipoles such as the (OH) group and in such cases they pack together so that dipoles of opposite polarity are in juxtaposition, as in the sugars and alcohols. In the organic acids and bases there are localized electrical charges which attract each other, as in inorganic ionic crystals, but here the shape of the molecule is the fundamental criterion of structure determining nature. For many years rubber was thought to be amorphous and possessing no ordered structure. We know it is a polymerized isoprene (C_5H_8). It is composed of "crystallites" or micelles which have dimensions of 600 by 500 by 150 Å. The interesting X-ray variations between unstretched and stretched rubber and rubber at room temperatures and at low temperatures are shown in Fig. 12.

Plastics, fibers, and rubbers are all organic high polymers, linear in their initial organization, which may be a permanent state or at least a preliminary state in their production. Polymers are made up of monomers, and when they occur in a string of only one monomer they appear as shown below:

(—M—M—M—M—M—M—M—M—)

If two or more different monomers exist, a copolymer results:

(—M—M—M—A—M—M—M—A—)

This process of forming polymers from monomers is known as polymerization. If in the process of polymerization a by-product, usually water, is formed, the plastic is said to be polymerized by condensation. In nearly all cases the reaction is induced or speeded by the presence of agents known as catalysts. These, of course, do not appear mixed in the final product. In general, the time required for polymerization is reduced by the application of comparatively low temperatures, about 350 F. being the maximum. When single bonds only are necessary to satisfy the elemental proportion the compounds are called saturated as would be the case with ethane (C_2H_6) as shown in Fig. 13. When double or triple bonding is indicated, the compounds are unsaturated as for ethylene (C_2H_4) and acetylene (C_2H_2). The benzene ring is an unsaturated compound with double bonds alternated with single bonds.

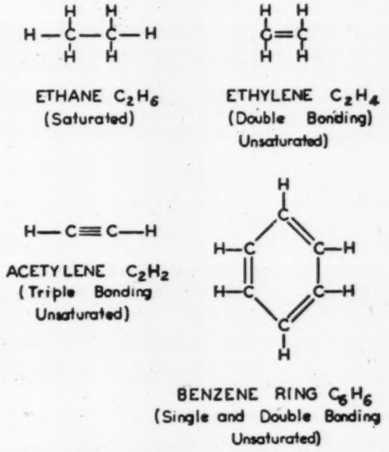
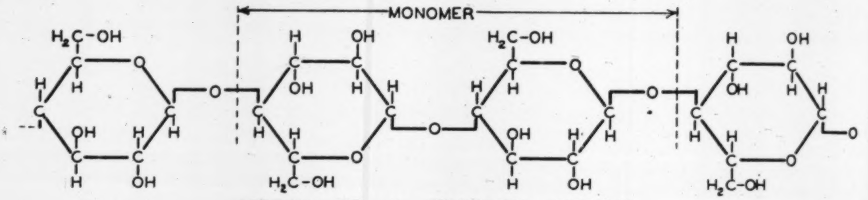
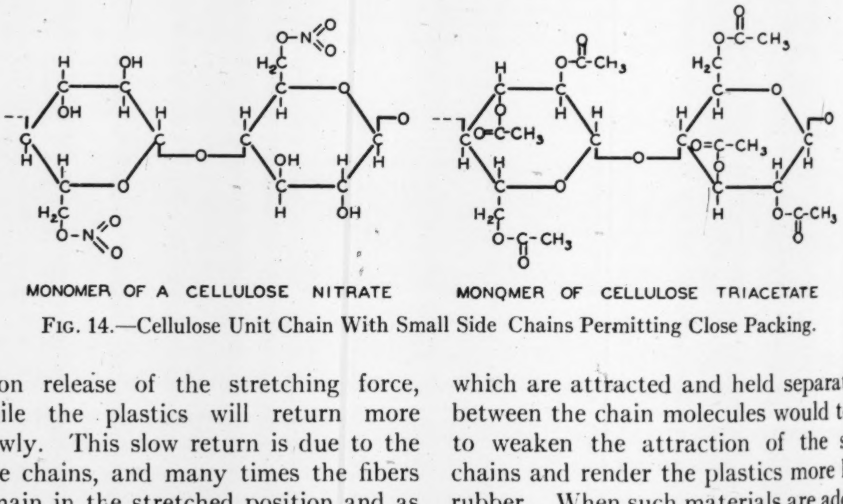


FIG. 13.—Different Types of Bonding and Degrees of Saturation.

In most high-polymer structures the straight chain carbon-to-carbon bonding forms the base of the structure, and the reactive portions of the molecules are found in side chains which branch from it. The important polymers, cellulose and cellulose derivatives and the phenolics, have rings as the major part of the backbone. The synthetic plastics, fibers, and rubbers being organic high-polymers, as well as the silicones which are primarily inorganic, can be differentiated if the molecular chains are arranged and extended along the same axis by extrusion or milling on rolls and then stretched. The rubbers generally will snap back to the original position upon release of the stretching force, while the plastics will return more slowly. This slow return is due to the side chains, and many times the fibers remain in the stretched position and as such will show a marked increase in tensile strength.



SECTION OF A CELLULOSE POLYMER



MONOMER OF A CELLULOSE NITRATE MONOMER OF CELLULOSE TRIACETATE

FIG. 14.—Cellulose Unit Chain With Small Side Chains Permitting Close Packing.

The structure of the side chains accounts for this difference in character. If the side chains are large and bulky and contain groups which exert only feeble attraction for groups on other chains, the chains cannot pack closely nor can they be held in a stable orientation when drawn by stretching, so the material snaps back to its original position. If the side chain groups are small and exert strong attractive forces, the material remains stretched, and the locking produced by the forces on these side chains between the closely-packed chains, increases resistance to slippage and rupture. Thus, by providing side chains with high bonding forces, we can increase strength and reduce stretch. Likewise, the introduction of substances which are attracted and held separately between the chain molecules would tend to weaken the attraction of the side chains and render the plastics more like rubber. When such materials are added they are known as "plasticizers" and have the function of rendering the plastics more soft and fluid when heated and more soft and flexible when cold. Thus, when the polymer chains are free and not interconnected, the material is fusible with heat and soluble in proper solvents. The material in this state is known as "thermoplastic." As bonds are formed between the polymer chains, the material becomes less readily softened by heat and more difficult to dissolve. A degree can be reached in the cross-linkage so that the material is practically infusible and insoluble. Such materials are known as "thermosetting." It is interesting to note here that heat speeds the reaction of cross-linkage but does not cause it.

Considerations of the cellulose derivatives such as cotton and wood fiber are interesting to illustrate the change from a practically infusible and insoluble material to one which is permanently fusible and soluble. The cellulose unit chain shown in Fig. 14 has small side chains permitting close packing. The reactive groups (OH) exert strong attraction between the chains. When cellulose is treated with a strong acid under proper conditions and with strong sulfuric acid present as a catalyst, the strong acid replaces the (OH) groups with the acid group, thus forming a salt, called an "ester." If this acid is (HNO_3), cellulose nitrate results; if acetic acid is used, cellulose acetate results; if a mixture of acetic and butyric acids is used, cellulose acetate-butyrate results. All of these are plastics, and all of them are permanently fusible and soluble, in marked contrast to cellulose. Another interesting interchange is indicated by the degree of nitration. When a cellulose nitrate contains 13 per cent N we have gun cotton; when it contains only 12 per cent N, we have a lacquer-base material; and when the percentage is reduced to 11 per cent we have a base for plastics. When camphor is used as a plasticizer we get the well-known plastic materials known as celluloid, Pyralin, and Nixonoid. These plastics are all easily dissolved or redissolved in organic solvents, except hydrocarbons, and have a medium water absorption. They tend to discolor and decompose when exposed for long periods to direct sunlight. The plasticizer is volatile, so in time the material becomes brittle. Cellulose acetate is used as a molding plastic, is permanently fusible and soluble, has relatively good resistance to sunlight, and rather poor resistance to water. When the fibers are spun from this plastic they are known commercially as "acetate rayon" or "Celanese" rayon.

The vinyl group shown in Fig. 15 is the base for most of the vinyl polymers. The mechanics of polymerization is the release of one of the double bonds giving each carbon a free bond to use linearly. This is by no means a simple reaction and requires very careful control of temperatures and careful consideration of the catalyst. When the third element in this monomer is chlorine (Cl) we have the vinyl-chloride base for polyvinyl chloride and polyvinyl chloride-acetate. When acetylene gas combines with hydrogen chloride, vinyl chloride is produced; when acetylene combines with acetic acid under the proper conditions, the product is vinyl acetate. These monomers are polymerized to produce polyvinyl chloride or polyvinyl acetate. The latter possesses exceptional adhesive qualities and is readily soluble in a wide range of organic solvents. It thus becomes a good surface coating and cement. Polyvinyl

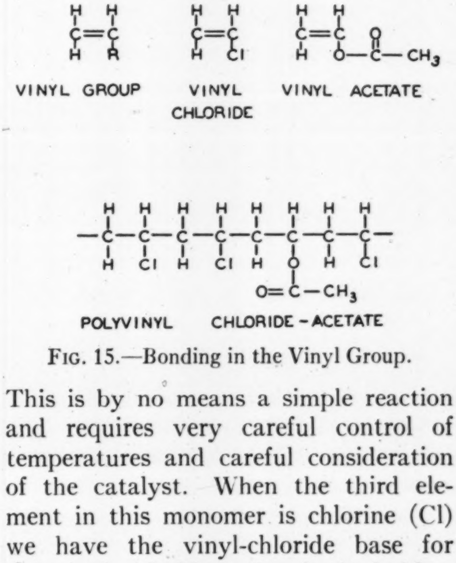
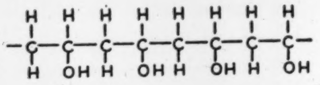


FIG. 15.—Bonding in the Vinyl Group.

chloride, although thermoplastic, is molded with great difficulty and is soluble only in high boiling point solvents. When vinyl chloride and vinyl acetate form a co-polymer, we have the results shown in Fig. 15. This material forms a good plastic and good coatings. It varies from a soft, rubber-like consistency to a rigid material suitable for electrical transcription records. It is often extruded around wire and it gives excellent electrical insulation and is permanently flexible, resistant to water, chemicals and ozone, and is noninflammable, due to the presence of the chlorine ion. When the polyvinyl acetate is treated with a strong water solution of caustic soda, under proper conditions, it is converted to polyvinyl alcohol, which is water soluble, and is the base for many of our paper coatings and cold-water paints. Its structure is shown in Fig. 16. Polyvinyl alcohol in turn may react with an aldehyde to form a polyacetal, the most important one of which is polyvinyl butyral. Since there are large bulky groups introduced into the side chains of the polymers, we may expect rubber-like properties. The material is too soft and rubbery for plastics, but it is tough, water-resistant and permanent. It is excellent material for waterproofing textiles and for the plastics sheet of safety glass. Thus, it has been used to produce "rubberized" fabric. A modified polyvinyl butyral, which cross-links partially when carefully heat treated and so does not soften upon reheating, is frequently used in safety glass. The bond of this material and its strength is indicated by the fact that a $\frac{1}{2}$-in. sheet of polyvinyl butyral with a $\frac{1}{8}$-in. thick plate glass on either side can resist the impact of a 15-lb. bird carcass when encountered by a plane travelling at 300 mph. The material is more permanently stable to sunlight and ozone than rubber. It is tough, flexible, and optically clear and transparent.


POLYVINYL ALCOHOL

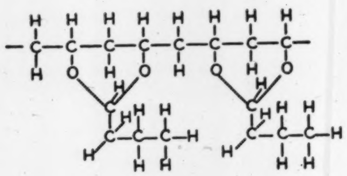
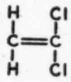

POLYVINYL BUTYRAL

FIG. 16.—Structures of Polyvinyl Alcohol and Polyvinyl Butyral.


VINYLIDENE CHLORIDE

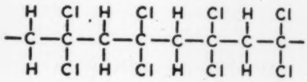

POLYVINYLIDENE CHLORIDE

FIG. 17.—Structure of Vinylidene Chloride and Polyvinylidene Chloride.

Vinylidene chloride (Fig. 17) is a monomer that can be used to form polyvinylidene chloride which is permanently fusible and soluble. It is very resistant to solvent action and is so difficult to control in fusion that it has not been found to be practical. When proportioned with vinyl chloride, forming a copolymer, and with other monomers for the purpose of controlling the action of the polymer, practical properties of considerable value are obtained. The vinylidene chloride and vinyl chloride copolymer is thermoplastic and can be molded in injection presses. It has been extruded as tubes up to 6 in. in diameter. As a fiber it has high tensile strength, good flexibility and permanence, but practically no stretch. This material is impermeable to water and water vapor, has excellent chemical resistance and is resistant to acids either strong or weak. Only one alkali, ammonia, affects it. It is resistant to almost all common organic solvents and has a softening temperature of 240 F.

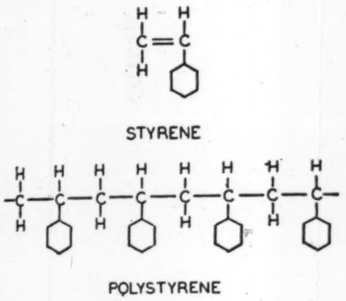


FIG. 18.—Formation of Polystyrene.

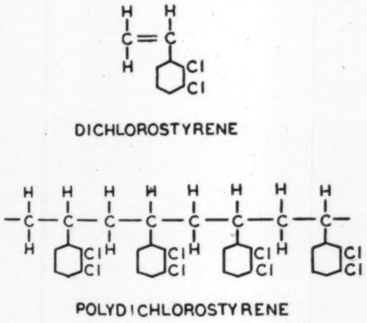


FIG. 19.—Structure Resulting from Substituting Two Chlorine Atoms for Two Hydrogen Atoms in Polystyrene.

The ethylene monomer shown in Fig. 13, and which is based on petroleum, reacts with hydrogen chloride to form ethyl chloride. When ethyl chloride reacts with benzene it produces ethylbenzene. The chemical decomposition of ethylbenzene by heat produces vinylbenzene, which is commonly called styrene. Figure 18 shows the chain structure of the styrene monomer in its complete form with a benzene ring and the abbreviated indication commonly used. When this monomer is combined to form a polymer it forms polystyrene (Fig. 18). This polymer is permanently fusible and soluble. It is transparent but turns yellow under the influence of light and possesses a very high refractive index. It has excellent resistance to water, acids, and alkalies, and has a low water absorption of only 0.04 per cent in 24 hr. of immersion. Presenting an outstanding resistance to the flow of electrical currents, particularly those of high frequency, it has a low dielectric loss factor almost the equivalent of fused quartz. It is inclined to be brittle, but when plasticizers have been used to relieve this situation many of the desirable electrical properties have been destroyed. Being a hydrocarbon it is naturally flammable, and it has a softening point below the boiling point of water. To correct some of these objections two chlorine atoms are substituted for two of the hydrogen atoms in the benzene ring forming the structure shown in Fig. 19 as a monomer and also as a polymer, the first being called dichlorostyrene, the latter polydichlorostyrene. When so treated, this polymer produces a material that is noninflammable, slightly less brittle and with a softening point of about 250 F. Its water absorption is reduced slightly and is very good. Its resistance to acids

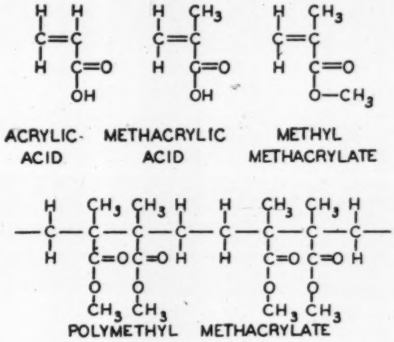


FIG. 20.—Transition of the Monomer Ethylene into a Succession of Subordinate Monomers.

and alkalies is excellent and its high-frequency insulation properties are good.

Figure 20 shows an interesting transition of the monomer ethylene into a succession of subordinate monomers, namely acrylic acid, methacrylic acid, and methyl methacrylate. It will be noted that if one of the hydrogens in ethylene is replaced by the organic acid group (COOH) that acrylic acid is formed. If a second hydrogen is replaced by the methyl group (CH_3) methacrylic acid is formed. The methyl salt, or ester, of methacrylic acid is methyl methacrylate, the monomer of an interesting polymer, namely, poly(methyl methacrylate), the structure of which is shown. This is the material commonly known by the name of Lucite or Plexiglas.

A brief consideration of the aldehyde condensation products is desirable. To produce these products the first step is to manufacture a heat-fusible resin suitable for molding; the second step is to convert this resin into an infusible material. The first stage consists of forming a linear polymer by the condensation reaction of an aldehyde with some other material. The second stage consists of the formation of cross-links between the chains by further aldehyde condensation reactions using catalysts. One of the principal aldehyde plastics is made with urea, which is produced from (CO_2) and (NH_3). Formaldehyde is produced from the methane in natural gas. The reactive parts of the urea molecule are the two amino groups, while in the formaldehyde the oxygen is the reactive portion. It is suggested that the action is one in which the amino group of the urea takes on a formaldehyde molecule, which later reacts forming water and the polymer. In the first stage the material can be used as an adhesive, and when this resin is dissolved in water it is called methylol or dimethylol-urea. Figure 21 shows the various steps for the production of the thermoplastic urea formaldehyde and the result of cross-linking in the presence of an acid catalyst forming the thermoset urea-formaldehyde.

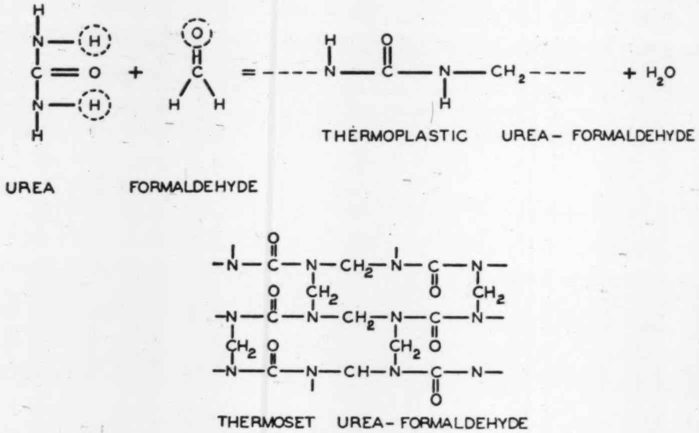


FIG. 21.—Steps in the Production of the Thermoplastic Urea Formaldehyde.

Besides the monomers discussed, we may list melamine and phenol which are used extensively to produce valuable formaldehyde products. The simplest phenol monomer which is the base of the "phenolics" is pure carbolic acid shown in Fig. 22. When this monomer reacts with formaldehyde, and the condensation product is catalyzed with an alkali or a small amount of acid, a product soluble in alcohol is produced. This solution is used for impregnating papers or fabrics to produce laminating adhesives. One of these, known as Novolac resin, is shown. The thermoset phenol-formaldehyde has a structure of somewhat different nature, as shown.

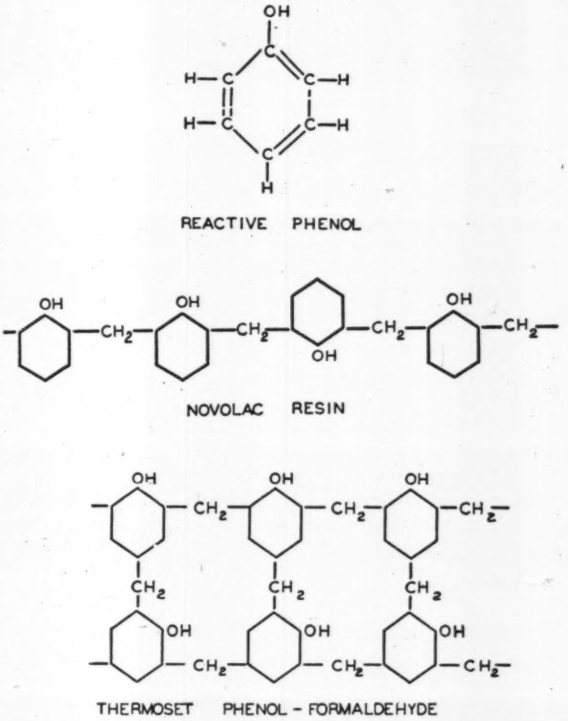


FIG. 22.—Structure of Phenol Monomer Which Is the Base of the "Phenolics"—Pure Carbolic Acid.

ADHESION

I am sure that by this time many of you have wondered why all this discussion of chemistry, physics, and crystallography. You may, however, be assured that a knowledge of the problem of bond between materials which are not homogeneous is an important element in the decision as to the desirability of many engineering laminates. It is with the question of adhesion that I should like now to deal.

In order that two compounds may form a complete or at least a substantial bond, or complete a series of substitutional solid solutions with each other, their chemical formulas must be analogous and they must have similar crystal structures in addition to the requirements imposed by the radii of the atoms from a geometrical point of view. We may speculate that if some of the active elements at the surface of a material which is to be bonded to another are compatible chemically or atomically, that we may have a chemical adhesion in the nature of ionic or homopolar bond. Where metals are involved, it would seem that it would be necessary that the electrons which are gyrating around the molecules must possess some attraction for the surface-active elements of the material to which it is to be attached, or the geometrical structure of one or the other should be such that they might have an opportunity to interlace. If the atoms of the adhesive are very small, they may fit into the interstices of even closely-packed structure. In metals particularly, they may thus form a solid solution without the necessity of substitution. It is thus possible perhaps that both substitution and interstitial solid solutions may exist in the same crystal or assemblage. A good example of this is in manganese steel in which the manganese atoms substitute for iron and the carbon atoms are interstitial. Mr. W. A. Weyl, in his very interesting paper on "The Theoretical Basis of Adhesion,"[3] has suggested something of the same sort in connection with his investigation with glass. He found that replacing the oxygen ion of a glass by other anions had a decisive influence on the adhesive properties. He found that in some cases it decreased the adhesion to a platinum crucible; in other cases where sulfur was substituted for oxygen, as in a phosphate glass, an adhesion to steel was produced. It seemed that the affinity of the anionic constituent of a glass to metal seemed to be the major factor of determining adhesion. This would indicate that adhesion is primarily a chemical phenomenon. Mr. Weyl claims that the gripping quality of the surface, although it might favor adhesion, contributes very little to the actual adhesion—an opinion which the author has held for quite a number of years.

The surface reactivity of most materials is shown by the adsorption of molecules from the atmosphere or other environment causing the saturation of surface residual forces. The adsorbed molecules contain groups which are active elements. In the case of glass and water these are OH groups which react and start a process of hydrolysis. Glass surfaces attract water and this hydrolyzes. The (O) ions are released and the (OH) groups extend above the surface. When the surface is approached by organic compounds, bonds are formed. Thus, animal glue will bond so completely to glass as to rupture the surface when the glue dries. The same phenomenon occurs when glass surfaces come into contact with organic silicones. Thus, when the reactive groups are kept under conditions which prevent their interaction, we may expect adhesive forces to act. In the case of water glass, which is highly polymerized, the Si-O-Na groups are responsible for adhesion. On the other hand, silicic acid or silica gel ages rapidly because of the free (OH) groups, which react readily with each other. This tendency of active groups to interact with each other, thus destroying their chemical reactivity, is responsible for the difficulties encountered in building new adhesives based upon chemical principles. The type of adhesion which involves such chemical surface action is often referred to as specific, as distinguished from the mechanical variety.

Mechanical adhesion is produced by interlocking. While there is no question but that penetration of the adhesive into the pores of an open face structure assists in maintaining bond, Browne and Truax[4] maintain that this mechanical effect accounts for only a small proportion of the joint strength in the case of wood. This seems to be borne out rather conclusively by the bond established by some adhesives with dense metal surfaces. If the penetrating tendrils of the adhesive were mainly responsible for bond, it would have to follow that the adhesive itself was highly resistant to tensile and shear forces. This we know is not true, in most cases.

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1506 (1946).

[4] E. L. Browne and T. R. Truax, "The Place of Adhesion in the Gluing of Wood," Colloid Symposium Monograph, Vol. 4, pp. 25 (1926)28–69.

It is quite likely that large ions, especially those which have incomplete or unsaturated outer electronic orbits, may change their shape and dimensions under the influence of the electrons of neighboring ions. This may cause concentrations of repulsion and subsequent attraction of considerable extent. There seems to be no question that there is a state of complete saturation, as conceived chemically, in the interior of a crystal, but there is some doubt whether this holds true for the surface. There is therefore a situation at the surface of all materials which would indicate that the ions cannot be completely surrounded by their partners. This leads, as Mr. Weyl says, to a "state of unsaturation." The phenomenon of surface tension and the tendency of small crystals to recrystallize to form larger crystals of smaller total surface area are good examples of this phenomenon. I shall not deal with Mr. Weyl's paper any further at this stage but I commend it to your careful reading together with the paper by Rinker and Kline on A General Theory of Adhesion, in the A.S.T.M. Symposium on Adhesives.[5]

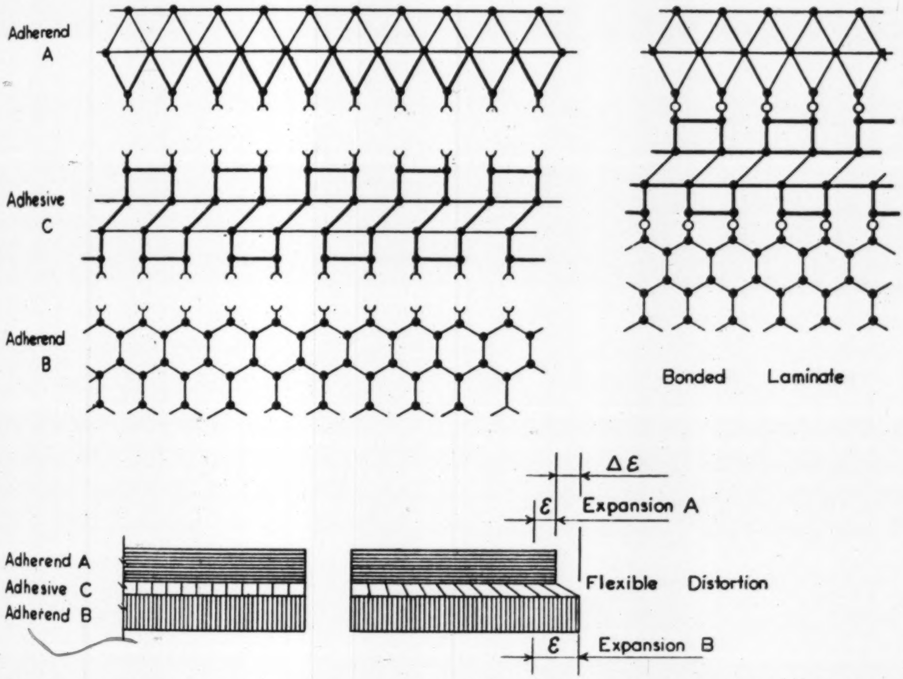

FIG. 23.—Diagrammatic Sketch of Bonding Two Different Materials Having Different Surface Structures.

> "Science carries us into zones of speculation, where there is no habitable city for the mind of man."
>
> —*Stevenson*

At this point I should like to step into the very conjectural field of hypothetical cases. Suppose we wish to bond a sheet of adherend A (Fig. 23) having a surface structure as indicated with a sheet of adherend B which has a slightly different surface structure. By surface structure I mean surface activity. To join these two surfaces we must have an interface adhesive or material which is compatible not only with the adherend A, but with the adherend B. We thus introduce this material, which is indicated as the adhesive C. Let us assume that the structure of the interface material is rhombohedral and thus deformable. We will apply the adhesive C to the adherend A and find that the surface structures coincide and produce a bond. Likewise, when adherend B is attached to the other side of the interface adhesive C, a similar compatible bond is produced. We now have a laminate which is nonhomogeneous. If we suppose that the thermal coefficient of adherend A is quite different from that of the adherend B, it becomes necessary for the bonded interfacial adhesive to be able to withstand the differential movement. The result of such differential movement is indicated in the figure. If, on the other hand, the interfacial adhesive or material has a structure which resists deformation without serious dislocation of the interatomic distances, it would be quite clear that the stress at the interface between the bonding adhesive and either adherend A or adherend B would be increased. This increase might be sufficient to cause a failure at the interface, and in many cases a very clean failure, even though the stress as measured might be quite high. In this case the adhesive is stronger than the bond. The result which might occur would be for the bond to be stronger than the adhesive so that it would be sufficient to resist the load imposed in shear but result in the complete rupture of the interfacial layer. It thus becomes quite evident that we must have a very clear conception—from a chemical point of view as well as from a physical point of view—of the relation between our adhesives and the various materials which we wish to adhere. How we may test these various situations is a

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[5] R. C. Rinker and G. M. Kline, "A General Theory of Adhesion," Symposium on Adhesives, Am. Soc. Testing Mats., p. 12 (1946). (Symposium issued as separate publication STP No. 65.)

TABLE IV.—NATURE OF BOND.

Bond		Adhesive	Type of Bond
Wood to	Metal	Rubber and phenolic resin	Hydrogen bridging between hydroxyl groups (wood and resin); sulfur bridging (rubber and metal); resin and rubber, complex structure.
	Wood	Phenolic resin urea-formaldehyde, casein, animal glue	Phenolic, hydrogen bridging hydroxyl groups; water molecules induce hydroxyl groups for others.
	Rubber	Rubber and phenolic resin	Hydrogen bridging (wood surface); interdiffusion (rubber surface).
	Plastics	Phenolic resin or compatible resin	Hydrogen bridging between hydroxyl groups. Interdiffusion at plastics face if thermoplastic. Hydrogen bridging at plastics face if thermoset.
Metal to	Metal	Solder, rubber, or resinous adhesive	Solder—forms mixed crystals or intermetallic compounds. Rubber—sulfur bridging resinous adhesives—through acid radical of the maleic acid copolymer of vinyl chloride-acetate.
	Vitreous enamels	....	Fusion dendrites penetrating and locking with enamel. Coordinate covalent bond.
	Rubber	....	Vulcanization—sulfur bridges.
	Plastics	Resinous adhesive	Acid radical copolymer compatible with metal and plastics.
Vitreous enamels to vitreous enamels		....	Fusion bond of like crystals.
Glass to plastics		Polyvinyl-butyral	Hydrogen bridging between hydroxyl groups due to hydrolysis of water on glass surface.
Rubber to plastics		Rubber solution with compatible plastic admixture	Interdiffusion and hydrogen bridging. (Rubber to rubber—strong rubber solution).
Plastics to plastics		Compatible resinous adhesive	Hydrogen bridging—reactivation of thermoset adherent. Interdiffusion and hydrogen bridging—thermoplastic adherent.

problem confronting every committee of the Society which tries to get at the fundamentals of failure in composite structures. Here we are again confronted, in the absence of basic information as to our proper approach, with the conduct of a series of tests conducted for the purpose of exploring these relations. If by such tests we get consistent results, as often we do, we have the right to set up a standard for the type of test required as related to the commercial use of the laminate. If, however, on the other hand, the results are incoherent, contradictory, and hard to explain, it would indicate very clearly that we have not solved the very fundamental relations that exist at the surface of the materials which are to be bonded, not to mention the type of material which we are using to produce this bond. In these cases our only escape is to make the structure so strong that it has a large factor of safety. This is the type of thing we are trying to avoid in order to reduce costs and conserve materials.

As the interfacial material is largely responsible for the nature of the bond, it is interesting to survey the proposed relation which exists when metals, enamels, rubbers, resinous adhesives, wood, and glass are bonded in some of the more common laminates. The information in Table IV is condensed in part from Rinker and Kline.[5]

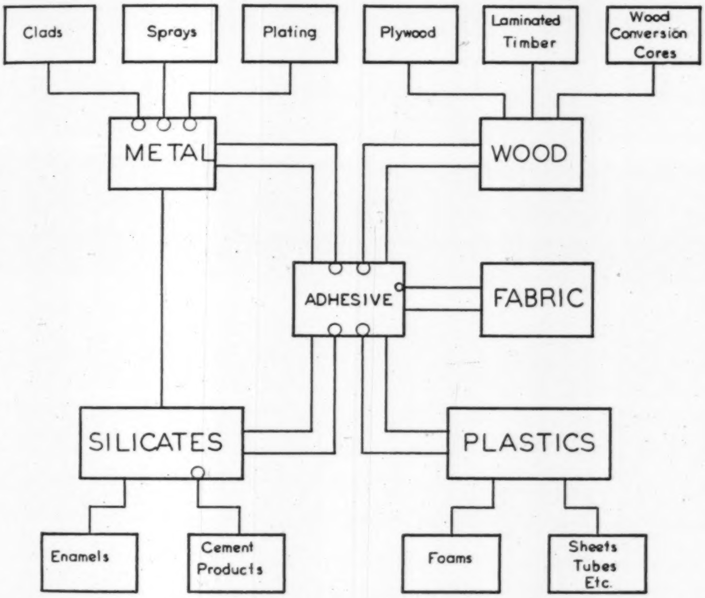


FIG. 24.—Possible Combinations for Laminates.

COMMON LAMINATES

The name "laminates" is used here to include those materials which require definite materials to effect adhesion and those combinations which involve adhesion by homopolar or metallic bond and the effects of interdiffusion. Figure 24 shows a chart which attempts to differentiate some of the more common engineering laminates and perhaps suggests others not as yet in common use. Thus metal, wood, fabric, plastics, and glasses can be combined with themselves and each other by the use of a proper adhesive. Metals can be clad with aluminum, magnesium, copper and copper alloys, nickel and nickel alloys, and several other metals; they are commonly sprayed with aluminum, copper, nickel, tin, zinc, and the like; they may be plated by electrolytic process with tin, zinc, copper, nickel, and the like; and they may be enamelled with quite a variety of silicate glasses and other base enamels. In these latter instances active interfacial adhesives are not used, and the adhesion depends upon surface activity and chemical bonds. Wood may be laminated in the various forms of plywood, laminated timber and the various wood conversion cores such as pulps, excelsiors, sawdust, fiber and the like with or without surface laminae of more dense and abrasion-proof materials. The silicates would also include cement-asbestos sheets with cement-asbestos foam cores, vermiculite mixtures and light-weight concretes such as aerocrete and others. The plastics may likewise be used as thermoplastic foams of great variety with plastic sheet, wood, metal, fabric or glass surfaces.

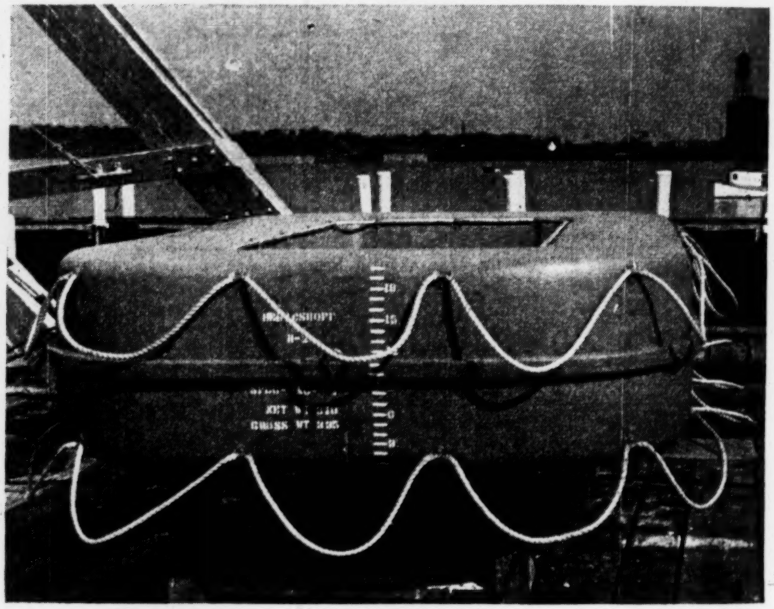


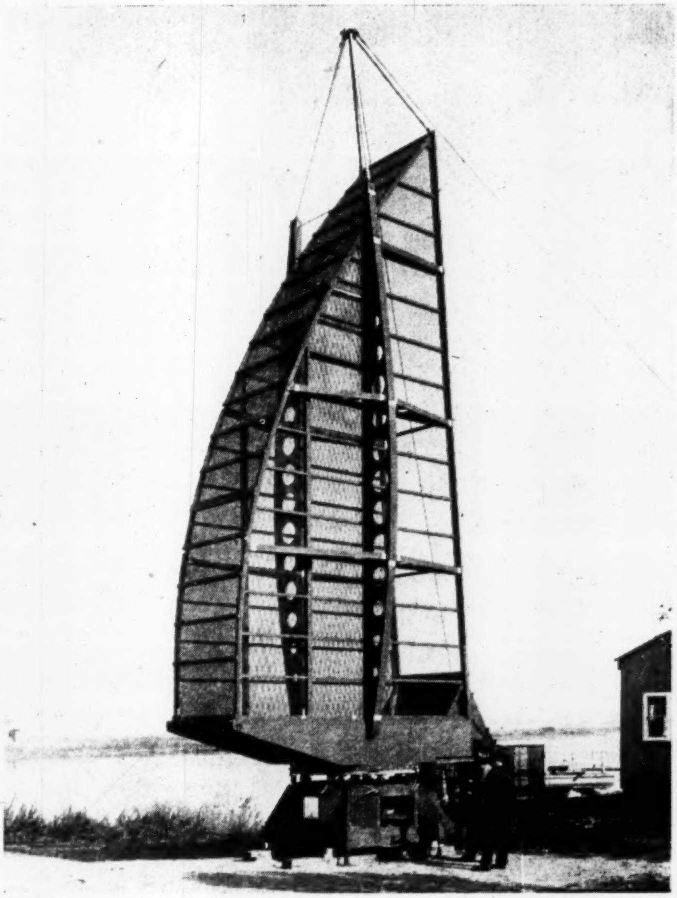
FIG. 25.—Twenty-Five Man Raft Made from Laminates.

Among the more commonly accepted laminates are those made of paper and fabrics. The fabric sheets may be of cotton, hemp, jute, flax, or fiberglass. Some are successive layers of wood formed with or without high pressures. Others are made up of metal and wood cores, cement asbestos with fiber-board core, plywood core faced with paper or fiberglass laminates, and fiberglass laminates or glass with honeycomb cores of various composition.

Besides some of the bimetals there are a great variety of "clads." These are steel sheets faced with thin gage aluminum, copper and copper alloys, magnesium and nickel and nickel alloys. These are in reality laminates and involve some of the same fundamentals of adhesion.

In the range of laminates made entirely of plastics, we find the laminated glasses and the polymethyl methacrylates such as Lucite or Plexiglas.

The great advantage of all of these materials is their susceptibility to forming, and this coupled with the possibility of providing surfaces which are resistant to many types of exposure and use make them extremely valuable in prefabricated assemblies. These assemblies vary all the way from flat sheets of considerable size to the most intricate shapes. It was this ease of simple shaping under low temperature and pressure and the great strengths which some laminates developed, that made possible some of the structures needed during the war. A few of these are shown in Figs. 25 to 27.



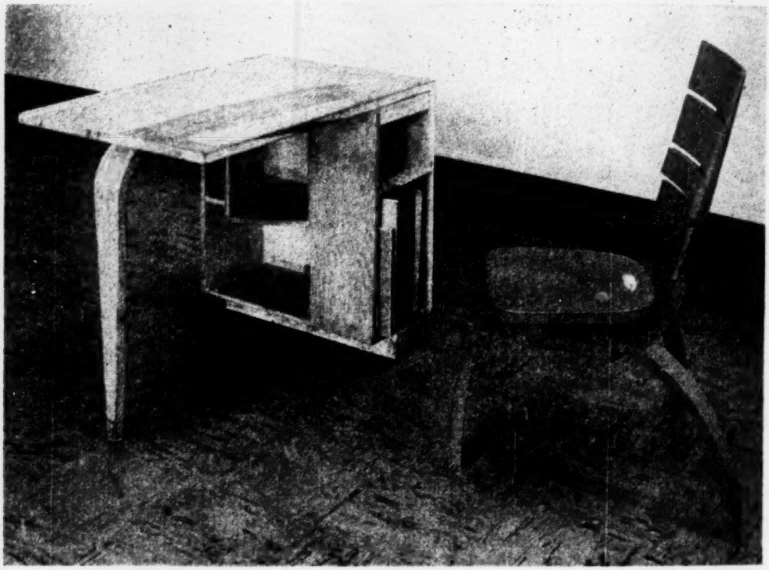
Courtesy Hagerty

FIG. 26.—Forty-Five Foot Radio Antenna, Largest Ever Built. Constructed from Wood and Expanded Sheet Aluminum.

The future of laminates is very bright and even in their infancy they have accomplished many amazing things. These laminates and the infinite composites still unknown, functioning in the way in which their earliest forerunner, plywood, has served so well, will speed prefabrication in the housing field, interior finishes, furniture, mechanical devices, and all of the endless variety of uses which synthesis introduces into this interesting field.

TESTING METHODS

The difficulties encountered in making tests of laminates are brought out by Platow and Dietz in their paper on Strength Properties in the A.S.T.M. Symposium on Adhesives.[6] With the permission of Messrs. Platow, Dietz and Koehn,[7] I have reproduced Figs. 9 and 10 of that paper (Fig. 28) which are excellent illustrations of the effect of the type of test specimen on the results which may be obtained. These figures show the use of rubber for the parts of the test specimens upon which equidistant white lines were spaced spanning over the bonded area. It will be noted that forces other than shear are acting in the single lap, double lap and double lap taper joints, and any conclusions drawn from this type of test without a knowledge of this condition would undoubtedly be erroneous. In making the actual tests, which in this case were for the purpose of determining the strength of the adhesive, it was hoped to get a bond with wood to wood in which a large percentage of failure was in the adhesive rather than in the wood. Just what a large percentage is or is not, is a matter of conjecture. If we could always get an adhesive that would cause failure in the wood, we could at least predicate our desired result on the strength of the wood. If the wood were a great deal stronger than was necessary for the particular function to be performed, it would be desirable to have an adhesive which would give us the desired shear value at all times but which would in no case fall below such desired value. This particular paper concerned itself only with impact strength, tensile strength, and shear strength between wood and wood, wood and metal, and metal and metal. It sug-



Courtesy Herreshoff Co.

FIG. 27.—Laminated School Posture Furniture.

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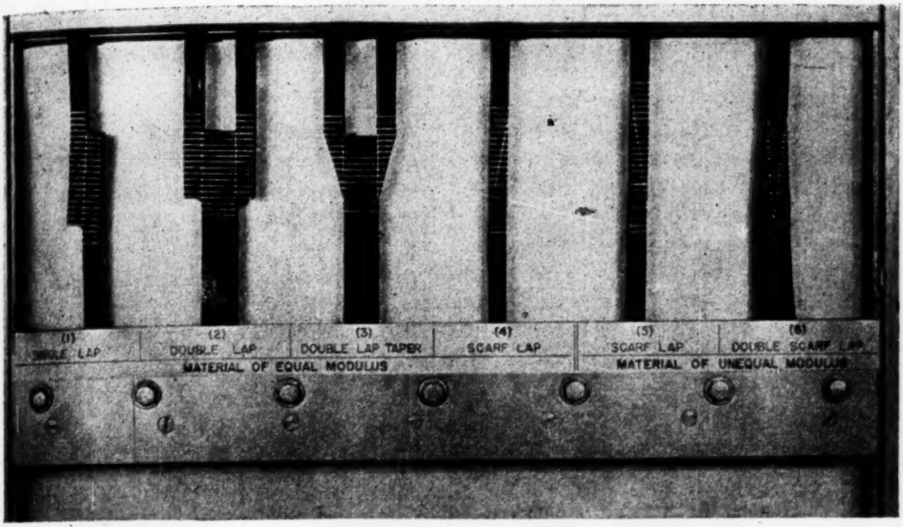
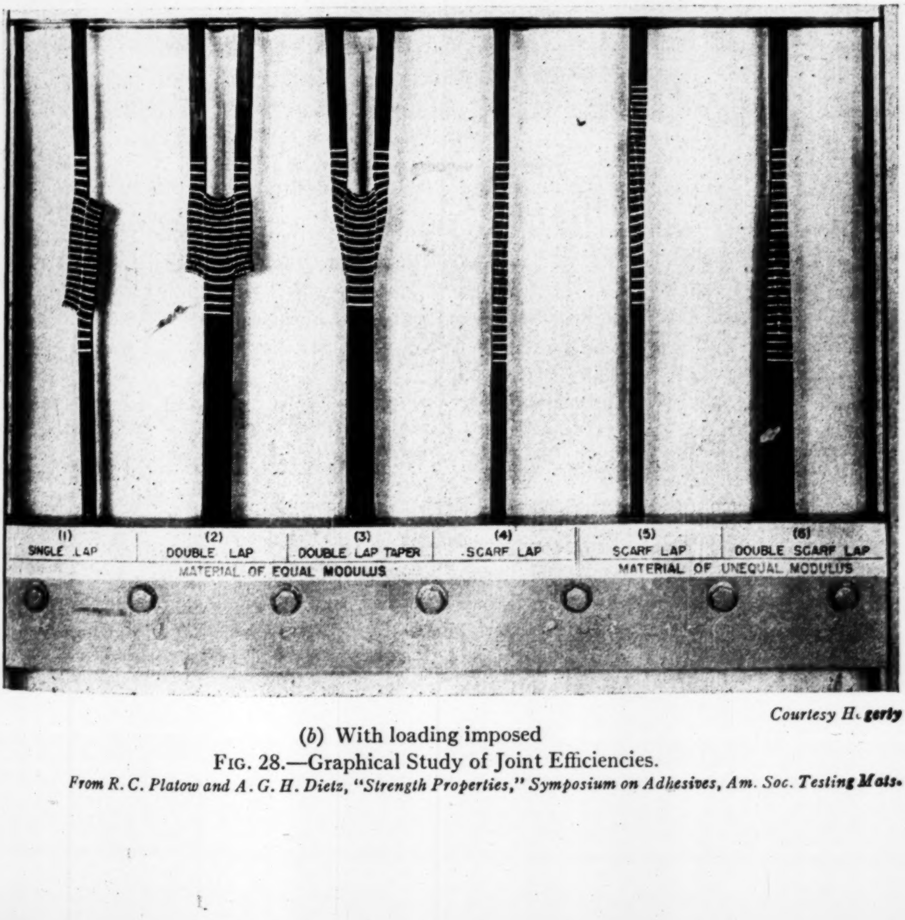
[6] R. C. Platow and A. G. H. Dietz, "Strength Properties," Symposium on Adhesives, Am. Soc. Testing Mats., p. 25 (1946). (Symposium issued as separate publication STP No. 65.)

[7] Armstrong Cork Co., Lancaster, Pa.


(1) SINGLE LAP
(2) DOUBLE LAP
(3) DOUBLE LAP TAPER
(4) SCARF LAP
MATERIAL OF EQUAL MODULUS
(5) SCARF LAP
(6) DOUBLE SCARF LAP
MATERIAL OF UNEQUAL MODULUS

(*a*) Before stress was applied


(1) SINGLE LAP
(2) DOUBLE LAP
(3) DOUBLE LAP TAPER
(4) SCARF LAP
MATERIAL OF EQUAL MODULUS
(5) SCARF LAP
(6) DOUBLE SCARF LAP
MATERIAL OF UNEQUAL MODULUS

Courtesy Hagerty

(*b*) With loading imposed

FIG. 28.—Graphical Study of Joint Efficiencies.

From R. C. Platow and A. G. H. Dietz, "Strength Properties," Symposium on Adhesives, Am. Soc. Testing Mats.

gests that various other strength tests such as flexural strength, the effect of peel, the effect of cleavage, creep tests, and methods of obtaining stress-strain data not only for these materials but for combinations of many of the others which are used commercially, should be made and studied. Fortunately many of these are now in progress. Regardless of what these tests may be, it is certain that the fundamental theory of adhesion must be reckoned with.

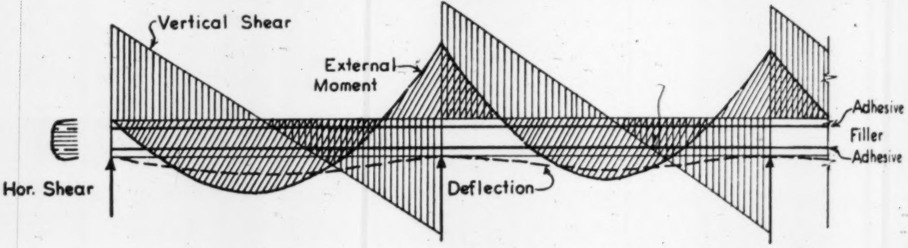


FIG. 29.—Conditions Which Exist in a Sheet of Laminate Under Flexural Load.

Let us explore the conditions existing in a sheet of laminate under flexural load. Taking the hypothetical sheet shown in Fig. 29 and assuming a uniformly distributed load, it might be assumed that the sheet would take the shape given. What are the forces acting in this case which we must consider and what are the characteristics of the various portions of this assemblage which must be present in order to resist such forces? It is very obvious that we must have sufficient tensile strength at the extreme fiber at the bottom so that the lower sheet will not part in tension. It is also equally obvious that the upper sheet should be sufficiently resistant to compression and crushing so that it will not fail at the upper fiber. Likewise, horizontal shear at the end supports must be resisted at its maximum by the filler material which might very well be some kind of plastic. This filler material must be able to transmit diagonal tension so as to allow the upper and lower sheets to act as a couple to resist the moment introduced by the loading. The additional strength that is contributed to a sheet of ordinary wallboard, which normally has low tensile and compressive strength, by the simple expedient of adhering a high tensile strength paper to the bottom and a veneer of high compression-resisting maple to the top, is amazing, provided that the end shears are taken care of by a shear resisting detail. In a flexural member of this kind, the shears at the interface between the filler material and the two sheets are practically uniform and very near the maximum, but it is extremely important that the material which forms the core of this laminate be carefully studied for shear values. It might be very interesting to take a section of this kind using rubber elements, and applying the technique used by Platow, Dietz, and Koehn and see what the stress distribution is. A study using rosette gages embedded at critical points would be illuminating. It would likewise be interesting to study this phenomenon by the means of photoelastic methods. We certainly must know something more about the distribution of stresses through the different types of core material and interfaces before we can deal at all with laminates which have high resistive tension and compression elements and low-strength core material. The strength of some of the bent sections of the wings of planes made during the war, which had a foam core usually of a urea type, is an illustration of what can be accomplished. In these instances, of course, the shape of the member contributed greatly to its strength. Nevertheless the distribution of stress through the core material will have to be studied a great deal before we know just what can be done with such structures. The war fostered many expedients of this type and most of them worked out very well, but as yet we do not know exactly why.

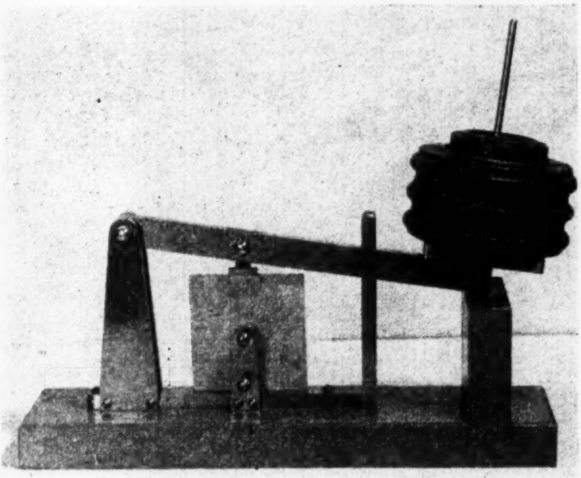


FIG. 30.—Apparatus for Testing Suitability of Plywood for Manufacture of English Mosquito Planes.

TABLE V.—PRESSURES TO SEPARATE FIVE-PLY PLYWOOD.

Lot	Condition	Specification	Face	Load per Linear Inch, lb.						Final Rating
				T	R	B	L	Avg.	Rating	
No. 1-1555-5	Dry	No. 9B	F1	130	85	121	82	104	G	G
			F2	115	109	100	133	89	G	
			B1	121	70	103	85	95	G	
			B2	124	145	112	82	116	G	
	Boiled	No. 2C	F1	64	55	70	40	57	P	P
			F2	100	94	100	115	102	G	
			B1	88	55	82	55	70	F	
			B2	58	94	64	55	68	F	
		No. 9D	F1	67	74	69	49	65	F	F
			F2	115	79	160	112	117	G	
			B1	82	61	85	52	70	F	
			B2	61	100	52	61	68	F	

The testing of cleavage resistance has been in the "cut-and-try" state. In the first place we shall have to define "cleavage." Is this phenomenon one of shear, compression, or tension, or do these stresses co-exist in varying proportion? It probably is some combination of two or all, depending upon the method of test. When confronted with the problem of testing the suitability of plywood for the manufacture of the English Mosquito planes, the author just reached into the hat,—all be it, a special hat,—and developed the apparatus shown in Fig. 30. It was hoped that this apparatus would give some sort of numerical comparative indices for acceptance. The knife blade was tapered from a razor edge to $\frac{1}{4}$ in. in a depth of $\frac{3}{4}$ in., and was 1 in. long. The razor edge was located as nearly as pos-

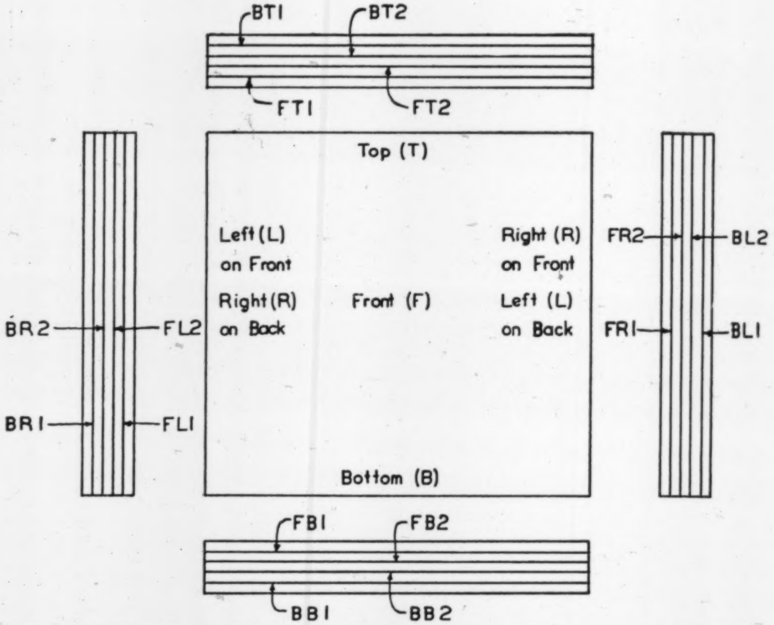

BT1
BT2
FT1
FT2
Top (T)
Left (L) on Front
Right (R) on Back
Front (F)
Right (R) on Front
Left (L) on Back
BR2
FL2
BR1
FL1
FR2
BL2
FR1
BL1
Bottom (B)
FB1
FB2
BB1
BB2

FIG. 31.—Schematic Drawing of Method of Testing Plywood with Apparatus Shown in Fig. 30.

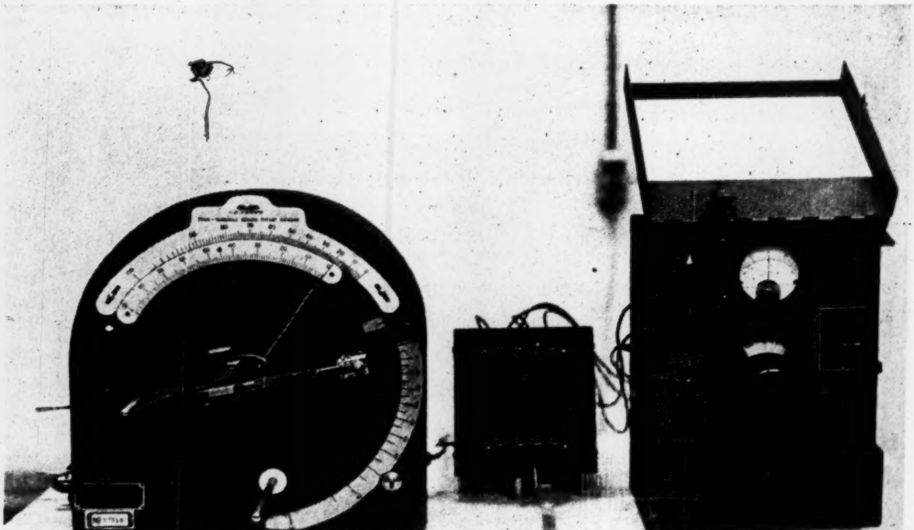


FIG. 32.—An Apparatus for Studying Creep in Plastics and Adhesives, Utilizing Ordinary Stiffness Tester, SR-4 Strain Gages, and a Modified SR-4 Strain Gage.

sible on the interface bonding material between the plies. The lever was loaded and the load which caused peeling or exfoliation was recorded. This process was repeated on each of the four edges of the square specimen and at each interface on each edge (Fig. 31). The tests were carried on with specimens in the dry state, and repeated with similar specimens after 3 hr. of boiling. Table V, taken from the report to the U. S. Treasury Department for whom the tests were made, shows a portion of the results obtained. Arbitrary values were assumed for minimum performance and the plywood was rated according to the following arbitrary scale:

Average Load per Inch			
≧ 85 lb.	Rating	G	(Good)
65 to 84 lb.	"	F	(Fair)
45 to 64 lb.	"	P	(Patchy)
<45 lb.	"	Pr	(Poor)

Final Rating = Lowest Joint Rating

If more than one individual test for any joint shows a value of <45, that joint should be rated Pr regardless of average load.

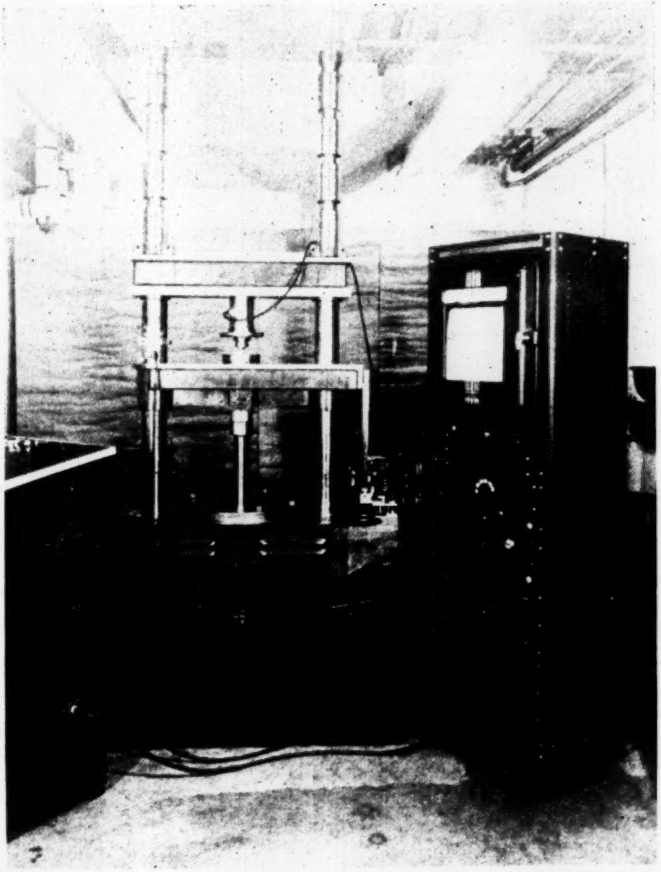


FIG. 33.—An Apparatus for Studying Creep in Plastics and Adhesives, Utilizing Servo-Controls to Give Constant Rates of Cross-Head Motion, Constant Rates of Load, or Constant Rates of True Strain, as May Be Desired.

It must be obvious to everyone that this approach is "hit or miss." Not only was the real phenomenon of bond not given the essential study for the particular adhesive used, but the method of test in itself assumed a combination of shear and tension which may not have been the proper criterion. Nevertheless, interesting information was obtained and shipments of plywood were tested by a mechanical device, instead of being left to the judgment of an inspector after a most superficial and variable test imposed by the English Government. Certainly the field of peel and cleavage offers a challenge to our ablest minds.

As for the tests already discussed, much must be done, perhaps by "cut and try," followed by a rationalization of approach based upon more scientific methods, before we shall understand the phenomenon of "creep," or plastic flow, particularly as applied to plastics and adhesives. This work will undoubtedly involve methods for obtaining stress-strain data.

Mr. Dietz has been busy developing apparatus to study this phenomenon for plastics.[8] Two of his machines are interesting applications of stress-strain studies. The first one, Fig. 32, utilizes an ordinary stiffness tester, SR-4 type strain gages, and a modified SR-4 strain recorder. Gages are mounted on the cantilever specimen, either directly or by means of paper backing, and the strains are observed as the specimen is loaded at various rates, to various stress intensities in the outermost fibers, and are held for varying lengths of time. For large strains the SR-4 recorder is modified by reduction shunts to reduce its sensitivity and to keep readings within the scale of the instrument.

The second, Fig. 33, is a universal testing machine in which servo-controls have been incorporated to give constant rates of cross-head motion, constant rates of load, or constant rates of true strain as may be desired. In plastics the time-temperature-strength interrelationship is so marked that it is necessary to be able to control with considerable accuracy the rates at which loads are applied over a rather wide range of speeds. The same holds true of composite materials—laminates and sandwiches—in which plastics or other time-strength sensitive materials are incorporated.

CLOSURE

I thus come to the end of our simple journey over the road of pure and applied research. I hope you have found at least certain areas interesting and stimulating. My fondest hope, however, is that I may have proven that the Society should continue its search for the properties of materials by applied research and that there will be some who will explore the "why" in areas of doubt. If our correlation of these efforts is extensive and if the search for "whys" is vigorous, we will make progress with assurance.

> "The development of modern science is secured not by the progress of any special branch of knowledge, but rather by the united efforts of various sciences and their mutual assistance in this striving forward."
>
> —*Sun Yat. Sen*

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[8] G. S. Brown, G. S. Burr, W. J. Gailus, L. D. Lipshutz J. O. Silvey, S. Yurenka, and A. G. H. Dietz, "Universal Plastics Testing Machine," see p. 1030.

A STUDY OF THE TRANSITION FROM SHEAR TO CLEAVAGE FRACTURE IN MILD STEEL*

By Harmer E. Davis,[1] Earl R. Parker,[1] and Alexander Boodberg[1]

Synopsis

Over ranges of temperature within which there occurred the transition from ductile shear-type failures to brittle cleavage-type failures, three types of specimens of three mild steels were tested: unnotched cylindrical tension bars, 3-in. wide flat bars with saw-cut notches in each edge, and 12-in. wide plates with a central notch at the midsection. The mechanism of fracture is discussed in relation to the variation in shear and cleavage strength with temperature and to the state of stress as induced by notching or deformation. Prestraining at a higher temperature was found to have an important influence on the cleavage strength which in turn affected the ductility at low temperatures. Depending upon the severity of the notch, through its influence on the state of stress and on the extent of plastic deformation before a crack is developed, the transition temperature of a mild steel may range from normal atmospheric to that of liquid air.

It has been recognized for some time that steel can fracture by two entirely different modes. Shear stress may cause slip, and also fracture, along certain crystallographic planes in iron and iron alloy crystals, while tensile stresses may cause cleavage fracture across a different set of crystal planes. The shear type of fracture is almost invariably associated with high ductility, while the tension or cleavage type of fracture may be preceded by a negligibly small amount of plastic flow. Differences in these two types of fracture were discussed in some detail in an earlier paper (1)[2] which dealt with the stress distribution in the necked section of a cylindrical tension bar of mild steel and the influence of temperature upon the type of fracture developed within the critical section.

The purpose of the tests reported in this paper was to extend the previously developed concepts to include an evaluation of the effect of plastic flow (prestraining) on the cleavage strength, and the influence of severity of notching on tendency toward cleavage fracture. The studies on prestraining were made on plain tension bars over a wide range in temperature. The notched-bar experiments were conducted on plates 3 and 12 in. wide. Three mild steels were included in the investigation, and to provide a basis for comparison, both of steels and of notch effect, standard Charpy tests were also made.

As the temperature of a ferrous metal is lowered, the yield strength (stress at which slip begins,—a shear phenomenon) increases; at a sufficiently low tempera-

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Associate Professor of Civil Engineering, Associate Professor of Physical Metallurgy, and Associate Professor of Mechanical Engineering respectively, University of California, Berkeley, Calif.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 499.

ture it may equal the fracture stress (2). The fracture strength as governed by excessive slip or detrusion also increases with drop in temperature, but not as markedly as does the yield strength. There is thus a tendency for decrease in ductility with lowering of temperature. The fracture strength as governed by cleavage depends upon the amount of slip or plastic flow which has previously occurred, being higher for greater amounts of flow up to some limit. If plastic flow is restricted, because of low temperature or by virtue of a state of stress in which the shearing stresses are small, the cleavage fracture strength may control the mode of failure, and owing to the limited plastic deformation, the material is said to become brittle. A discontinuity, such as a notch, in a stressed body creates a stress condition which inhibits plastic flow and thus cleavage fractures can occur at higher temperatures in notched bars than in unnotched bars. Further, the more severe a notch in effect, the higher is the temperature at which cleavage fractures can develop. Conversely, for a given material, the temperature at which cleavage fractures occur, may serve to evaluate the degree to which the individual state of stress is effective in restricting plastic flow.

In this investigation a characteristic of primary interest is the range in temperature above which a material fails entirely by shear, and below which only cleavage fractures develop. This is designated as the transition temperature range. Within the transition temperature range a mixture of the two modes of fracture occurs.

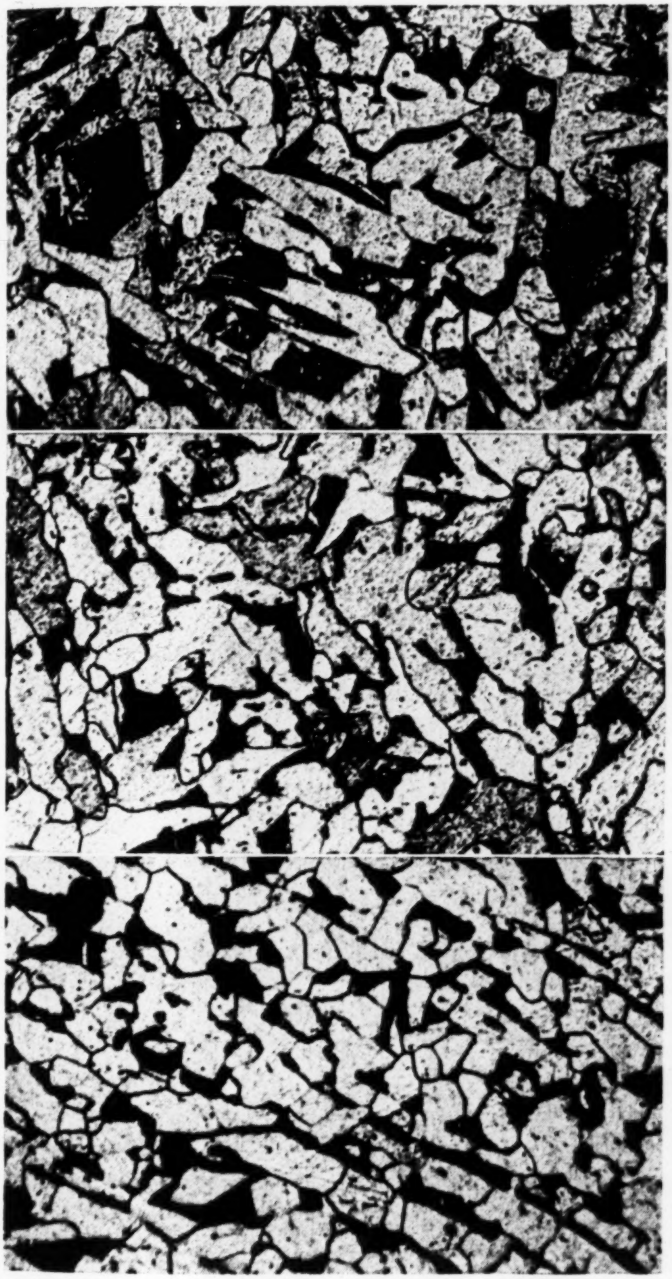
When, in a given series of tests, the temperature is varied over the range within which transition in mode of fracture takes place, as cleavage fractures occur over increasing percentages of the surface of rupture, there is a corresponding lesser amount of total plastic deformation up to the point of complete failure; this is reflected in a decrease in total energy absorbed. Indeed, it is this correlation between absorbed energy and restriction in plastic deformation which has made the Charpy test such a useful indicator of the relative tendency of similar metals toward notch embrittlement.

TABLE I.—COMPOSITION AND MECHANICAL PROPERTIES OF STEELS.

All steels of semi-killed class. All steels tested in as-rolled condition.

	Steel A	Steel B	Steel C
Chemical Composition, per cent by weight			
C	0.26	0.18	0.24
Mn	0.50	0.73	0.48
Si	0.03	0.07	0.05
P	0.012	0.008	0.012
S	0.039	0.030	0.026
Ni	0.2	0.05	0.02
Al	0.012	0.015	0.016
Cu	0.03	0.07	0.03
Cr	0.03	0.03	0.03
Mo	0.006	0.006	0.005
Sn	0.003	0.012	0.003
N	0.004	0.005	0.009
At Ladle			
Deoxidation Treatment, additions per ton of steel	Ferro-Silicon: 1.3 lb.	Ferro-Manganese: 8.5 lb. Ferro-silicon: 1.1 lb. Aluminum-silicon: 2.5 lb.	Ferro-Manganese: 6.0 lb. Ferro-silicon: 2.6 lb.
At Mold			
Physical Properties	Aluminum: 0.5 lb.	Aluminum: small amount	Aluminum: 0.3 lb.
Yield Point, psi	35 500	32 000	35 500
Tensile Strength, psi	58 500	56 500	63 500
Elongation, per cent in 2 in.	43.0	44.5	42.0

Observations made in these and related series of experiments have indicated that the percentage of the fracture surface which fails by cleavage (or by shear) may be used as a basis for determining the transition temperature range, although the limits are not always defined as precisely as by measurement of the energy absorbed to rupture. In some of the tension tests, where the strain measurements necessary for the calcula-



Steel A Steel B Steel C

FIG. 1.—Photomicrographs of Steels A, B and C (X100).

tion of energy would involve a large expenditure of time and effort, the percentage-of-shear fracture basis was used to advantage.

MATERIALS AND METHODS OF TESTING

The materials used in these studies were three mild steels of the semi-killed class, designated herein as steels A, B, and C. Compositions and mechanical properties, as determined by standard tests, are given in Table I. The microstructures of these steels are shown in Fig. 1.

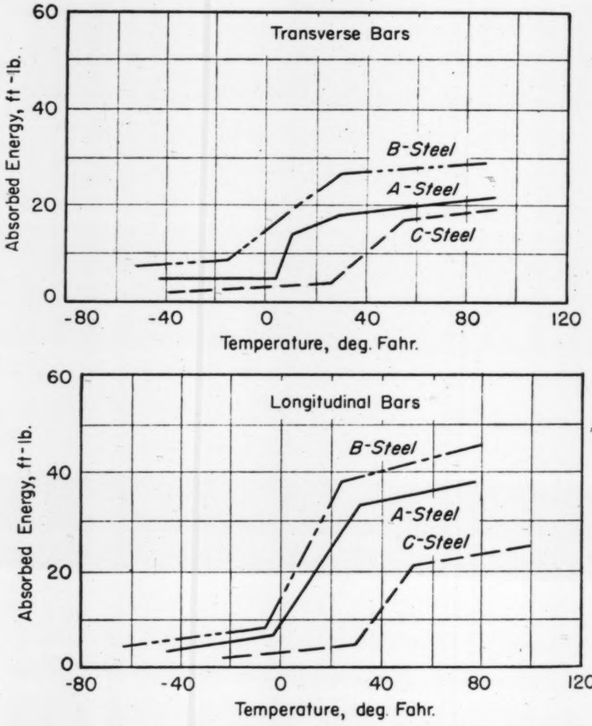


FIG. 2.—Typical results of Charpy Impact Flexure Tests for Steels A, B, and C.

Notch—standard keyhole type
Longitudinal bars have long axis parallel with direction of rolling of plate; transverse bars have long axis perpendicular to direction of rolling of plate; all notches were perpendicular to direction of rolling and to face of plate.

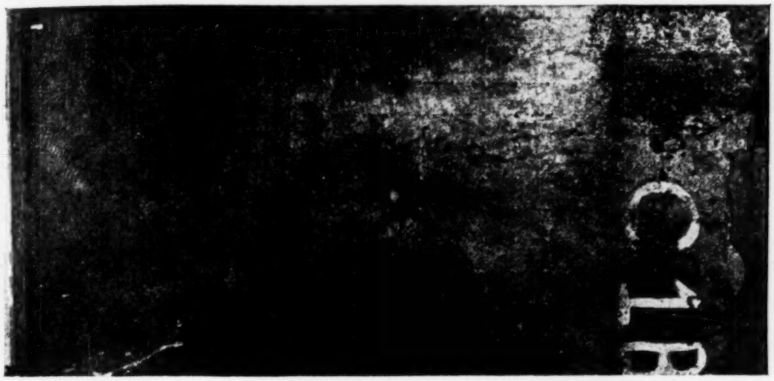
The relative tendencies of the three steels toward notch embrittlement as indicated by standard Charpy impact tests on specimens with keyhole-type notches is given in Fig. 2. Steel C exhibited the highest, steel A a somewhat lower, and steel B the lowest transition temperature. The steels A and C had approximately the same composition, and although their microstructures differed somewhat, it is believed that this difference was not sufficiently great to account for the disparity in their transition temperatures; a possible cause of the difference was the nitrogen content, that for steel C being about twice that for steel A.

In addition to the standard tension and Charpy specimens used for classifying the materials, three special types of specimens were used in the tests:

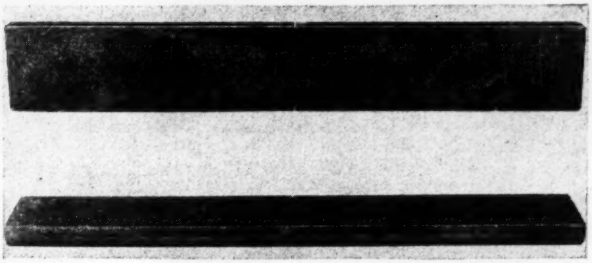
1. Cylindrical tension bars, similar to

standard tension specimens, but having a diameter of $\frac{3}{8}$ in.

2. Flat plates (for testing in tension), 12 in. wide by $\frac{3}{4}$ in. thick, cut from as-rolled plate, with a notch or slot cut in the center of width of the plate at the midsection. The slot was made by drilling a $\frac{1}{2}$-in. hole at the center of a plate, making two straight hacksaw cuts outward from opposite edges of the hole for a distance of $1\frac{1}{4}$ in. toward the edges of the plate, and then extending these cuts for an additional $\frac{1}{8}$ in. by means of a jeweler's saw having a blade 0.010 in. thick. The transverse notch thus formed occupied one quarter of the width of the specimens. The appearance of these specimens is shown in Fig. 3(*a*).



(*a*) 12 by ¾-in. Flat Tension Specimens with Central Notch.



(*b*) 3 by ¾-in. Flat Tension Specimens with Edge Notch.

FIG. 3.—Flat Bar Specimens Before Test.

3. Flat tension bars, 3 in. wide, with notches in each edge at the midsection cut to a depth of $\frac{1}{4}$ in. by means of a hacksaw. In one series of tests the specimens were $\frac{3}{4}$ in. thick, cut directly from as-rolled plate of each of the three steels. The appearance of these specimens is shown in Fig. 3(*b*). In a second series of tests, specimens cut from $1\frac{1}{8}$-in. plate of steel C were machined to various thicknesses; tests were made on specimens having thicknesses of $1\frac{1}{8}$, 1, $\frac{3}{4}$, $\frac{5}{8}$, and $\frac{1}{2}$ in.

Tests were conducted at various temperatures ranging from atmospheric down to -300 F. In making the tension tests at temperatures other than atmospheric, the specimens were enclosed in small cooling chambers during test. The desired temperatures were obtained within the cooling chambers through the use of air or alcohol, cooled by dry ice, or through the use of liquid nitrogen. The flow of the coolant was controlled manually, and the temperatures were measured by means of thermocouples either soldered to the surface of a specimen, or wrapped tightly against the surface and covered with an insulating pad. The design of the cooling chambers and the method of circulating the coolant was such that the temperature varied only a few degrees over the effective length of specimen.

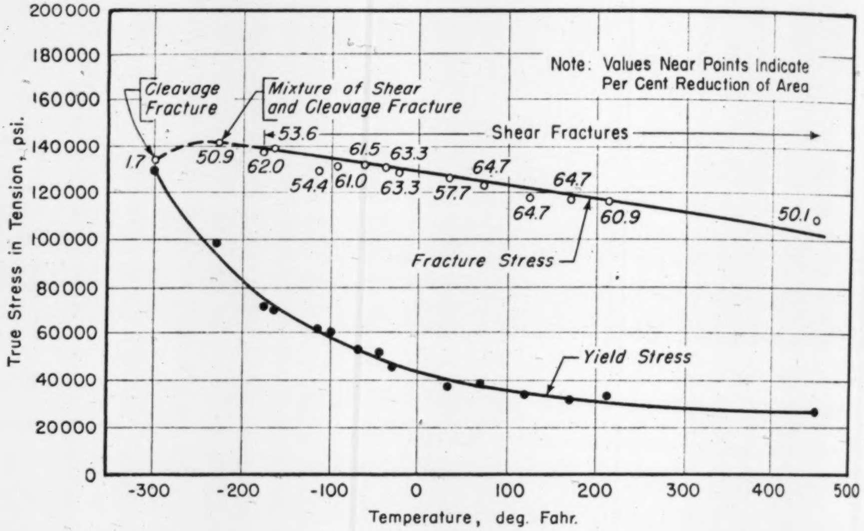


FIG. 4.—Variation of Yield and Fracture Stresses with Temperature—Steel A.
C = 0.26 per cent, Mn = 0.50 per cent
Specimens: ⅜-in. diameter tension bars.

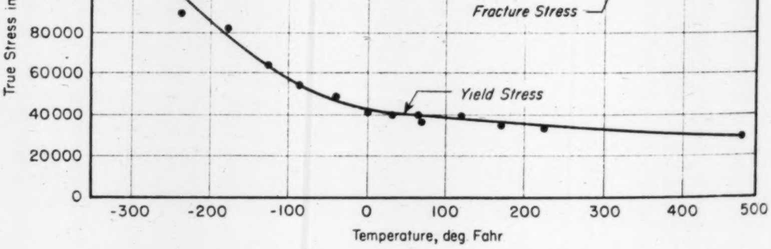


FIG. 5.—Variation of Yield and Fracture Stresses with Temperature—Steel B.
C = 0.18 per cent, Mn = 0.73 per cent
Specimens: ⅜-in. diameter tension bars.

The rate of straining was slow in all cases. The time required to load the tension specimens to fracture ranged from one half to several hours.

RESULTS OF TESTS

Plain Unnotched Tension Bars:

The variations with temperature of the strength and ductility of plain cylindrical tension bars made from steels A, B, and C are shown in Figs. 4, 5, and 6, respectively. The ordinates to these diagrams are "true" stresses, that is, axial load divided by actual cross-sectional area at a given load. The numerals adjacent to the points along the fracture-stress diagram give the reduction in area in per cent. The type of fracture is also indicated. At the lowest temperature of test (about −300 F.), the unnotched specimens of each of the steels failed by cleavage after but little plastic flow. As the temperature of test was dropped below about −250 F., the fracture strength decreased; this is attributed to a lesser amount of plastic flow occurring below this temperature. Two of the steels, B and C, after yielding slightly, fractured at stresses less than the yield strengths. The appearance of a series of fractured specimens from this group of tests is shown in Fig. 7.

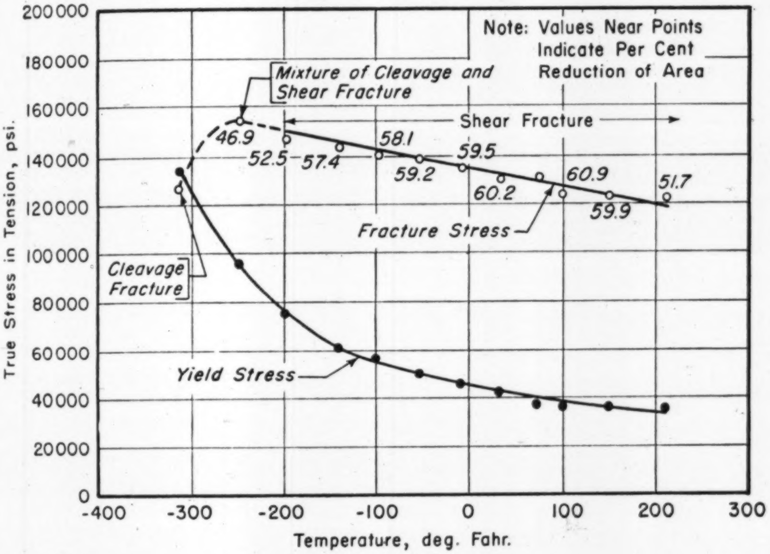


FIG. 6.—Variation of Yield and Fracture Stresses with Temperatures—Steel C.
C = 0.24 per cent, Mn = 0.48 per cent
Specimens: ⅜-in. diameter tension bars.

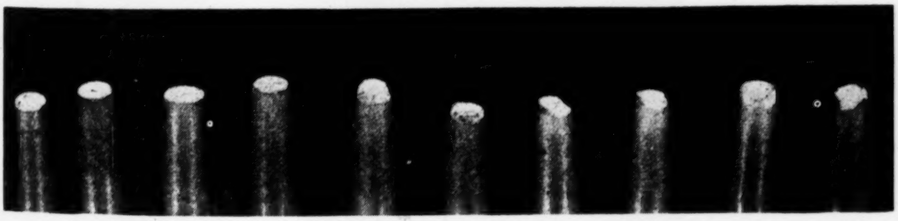


FIG. 7.—Appearance of Fractured ⅜-in. Diameter Tension Bars Tested at Various Temperatures.

The study of the influence of plastic flow, prior to rupture, upon the cleavage fracture strength was undertaken in a series of tests on similar plain tension bars of steel A, the results of which tests are summarized in Fig. 8. In these experiments, at each of three temperatures (−110, 70, and 212 F.), specimens were strained to obtain various reductions of area up to 50 per cent and then cooled immediately to −300 F. and loaded to failure. No additional plastic flow was observed at the low temperature. In Fig. 8, in which the true stresses at fracture at −300 F. are plotted against the temperature at which the test specimens were prestrained, the marked effect of cold working upon the cleavage strength is apparent. While with no prestraining the fracture strength was 133,000 psi., for a 50 per cent reduction of area the fracture strength was of the order of 195,000 psi. The temperature at which the prestraining took place appeared to have little effect, if any, upon the fracture strength.

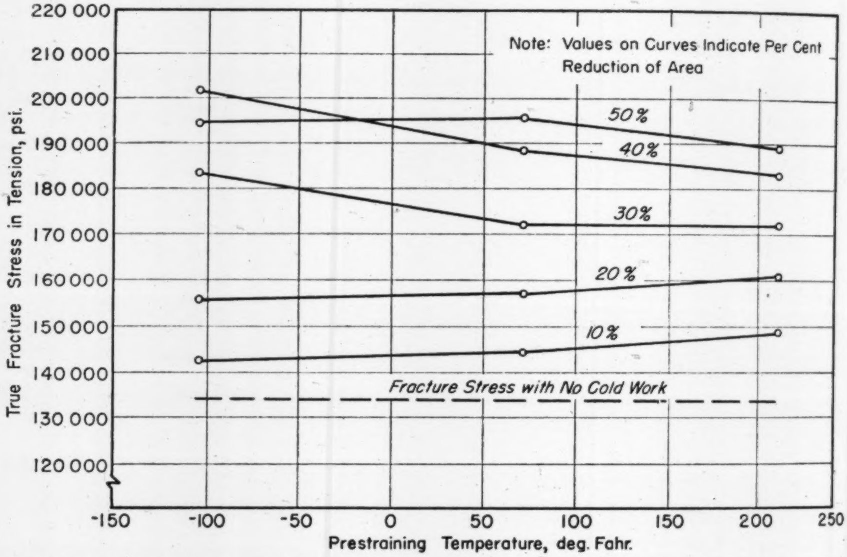


FIG. 8.—Effect of Prestraining at Various Temperatures on Cleavage-Fracture Stress at −300 F.

Twelve-Inch Flat Plates:

Results of the tension tests of the 12-in. wide by $\frac{3}{4}$-in. thick centrally-notched plates of each steel are given in Figs. 9 and 10. In these experiments extensometer measurements were made at each temperature of test so as to obtain data from which the energy absorbed during loading could be computed. For these specimens it was found that the energy absorbed to maximum load was a better criterion on which to base estimates of the transition temperatures than was energy to complete separation of the parts of a plate, because at the higher temperatures the extreme distortions of the specimens after a major portion of the separation had occurred resulted in drastically-changed stress conditions. Plots of energy absorbed to maximum load *versus* temperature for each of the steels are shown in Fig. 9. The transition temperatures were highest for steel C, lowest for steel B, with that for steel A intermediate between the other two—the same relative order as obtained in the Charpy tests. The range in transition temperature as indicated by the percentage of the area of fracture in shear is shown in Fig. 10.

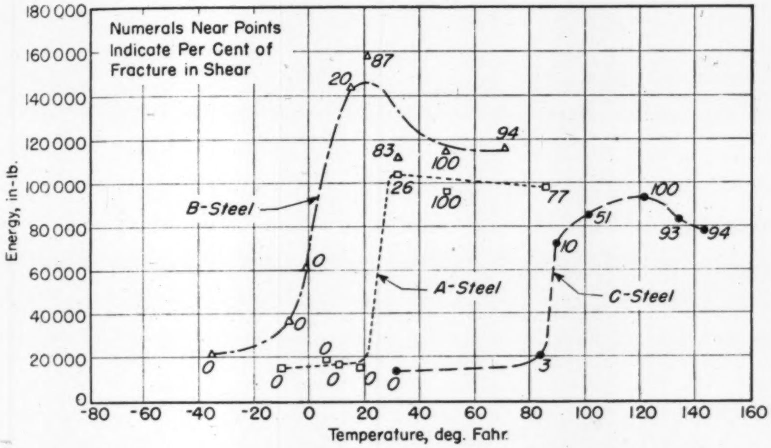


FIG. 9.—Variation with Temperature of Energy Absorbed to Maximum Load for 12-in. Wide Notched Plates in Tension.

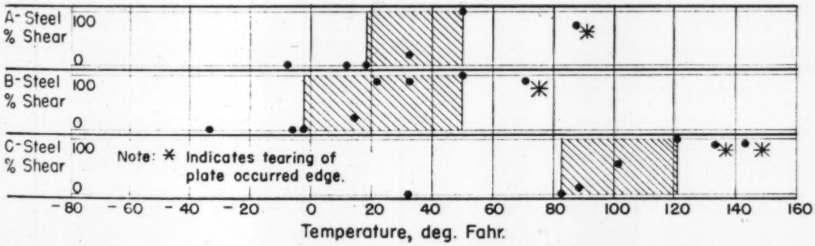


FIG. 10.—Variation with Temperature of Percentage of Shear-Type Fracture in 12-in. Wide Notched Plates in Tension.

Stress conditions greatly altered during fracture by tearing of plate near one edge.

Three-Inch Flat Bars:

Results of the tension tests on 3-in. wide, edge-notched flat bars are shown in Figs. 11, 12, and 13. In the tests of these specimens, the energy to fracture was not determined, but the transition temperature range was obtained by measuring the percentage of the area of fracture in shear. Plots indicating the transition temperatures for the three steels as determined by tests on $\frac{3}{4}$-in. thick specimens of full plate thickness as-rolled are given in Fig. 11. Photographs showing the appearance of the fracture surfaces of specimens in this group of tests are reproduced in Fig. 12. The tests also placed the steels in the order as regards notch sensitivity as did the tests on Charpy bars and 12-in. flat plates.

To determine the effect of thickness of plate on the transition temperature,

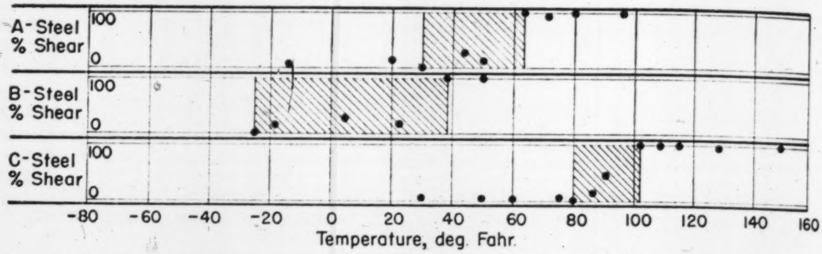
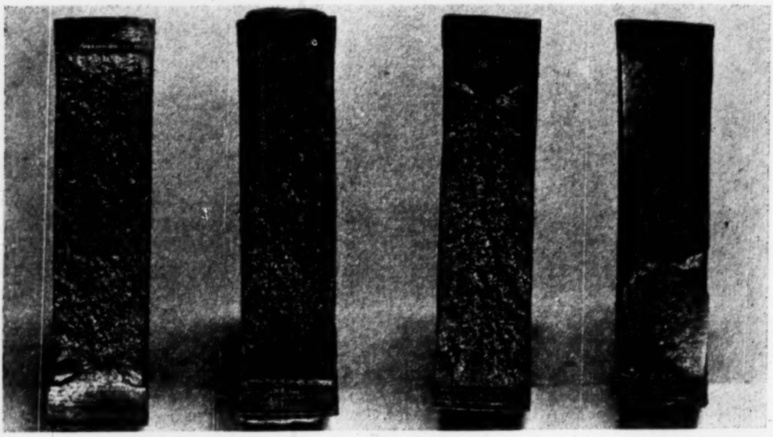


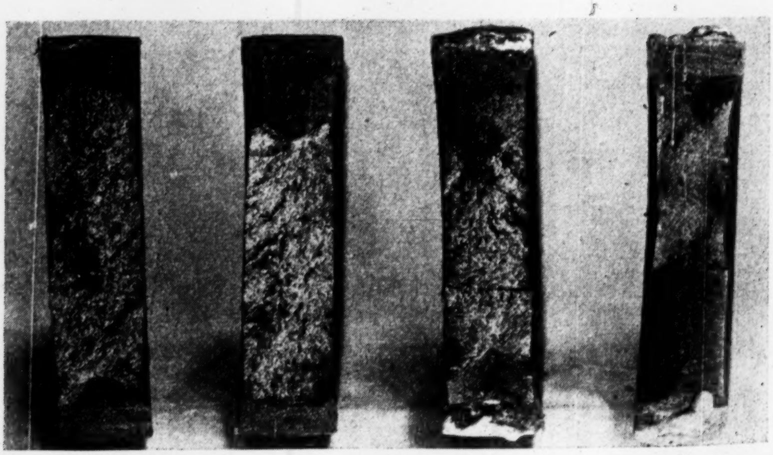
FIG. 11.—Variation with Temperature of Percentage of Shear-Type Fracture in 3-in. Wide Notched Bars in Tension.

All bars ¾-in. thick from plate as-rolled.



Temperature, deg. Fahr..	—15	30	51	72
Shear, per cent.........	3	2	10	97

Steel A



Temperature, deg. Fahr..	19	6	22	39
Shear, per cent..........	6	16	39	100

Steel B

FIG. 12.—Fractured 3 by ¾-in. Edge-Notched Specimens, Showing Effect of Temperature on Type of Fracture.

The portions of the fracture surface having the coarse-textured, granular appearance are in the cleavage mode. The portions of the fracture surface having a relatively more fine-textured appearance are in the shear mode.

3-in. wide bars having thicknesses ranging from $\frac{1}{2}$ to $1\frac{1}{8}$ in. were machined from $1\frac{1}{8}$-in. plate of steel C. The results, based upon measurements of percentage of shear fracture area are summarized in in Fig. 13. The effect of increased thickness in producing a more severe notch effect is apparent from the trend toward higher transition temperatures for the thicker plates.

DISCUSSION

The development of a workable picture of the mechanism of failure in mild steel requires recognition of the following factors: (*a*) the two modes by which fracture can occur, that is, by excessive slip due to shearing stresses, and cleavage separation due to tensile stresses, (*b*) the variation in the yield strength, the shear fracture strength, and the cleavage fracture strength with temperature, (*c*) the possibility that one or another mode of fracture may be induced at a given temperature depending on whether the shearing or the tensile stresses become critical in relation to the shearing or tensile strengths—a condition governed by the state of stress at the point of incipient failure. The general way in which these factors are interrelated has already been clearly outlined by Gensamer (2). The results of these experiments substantiate, quantitatively, Gensamer's explanation; in addition, an evaluation of factors such as the influence of plastic flow on the cleavage strength is provided by the data.

The strength characteristics of the steels change with temperature, as shown in Figs. 4, 5, and 6. The yield strength of steel A, for example, increases about 500 per cent as the temperature is lowered from about 400 to −300 F. The fracture strength as governed by shear, also increases as the temperature is lowered, but the change in magnitude is less, over the range of temperature investigated, than the change in magnitude of the yield strength. It might be concluded from this that the ductility of the materials would progressively decrease as the temperature is lowered, but an examination of the values of percentage reduction of area indicates that no appreciable change in ductility occurs

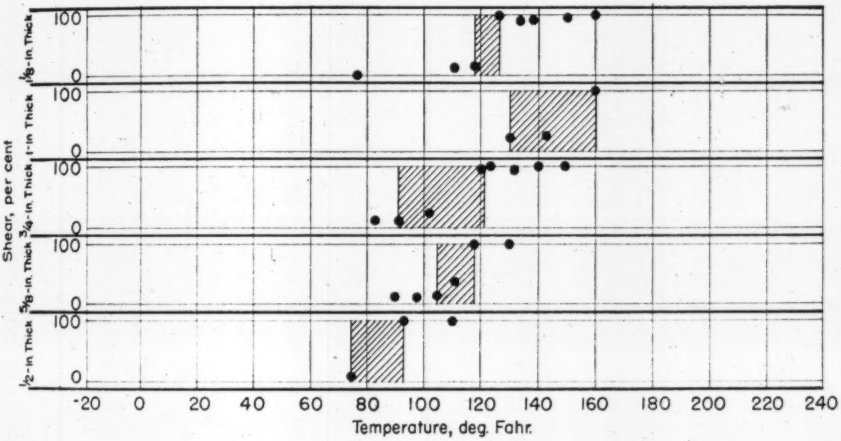


FIG. 13.—Variation with Temperature of Percentage of Shear-Type Fracture in 3-in. Wide Notched Bars of Various Thicknesses.

Material: steel C
Specimens machined to given thickness from 1⅛-in. plate.

over the range in which *shear* fractures take place, that is, down to about −200 F. in this case.

As the temperature is reduced below −200 F. for these steels, the yield strength increases rapidly, the ductility decreases, and the fracture surfaces of broken bars show increasing areas having a coarse crystalline texture associated with cleavage.

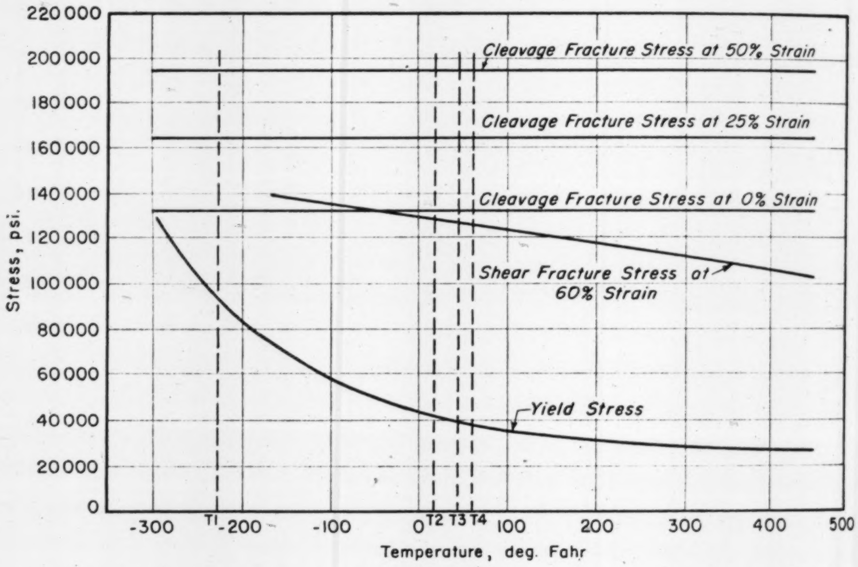
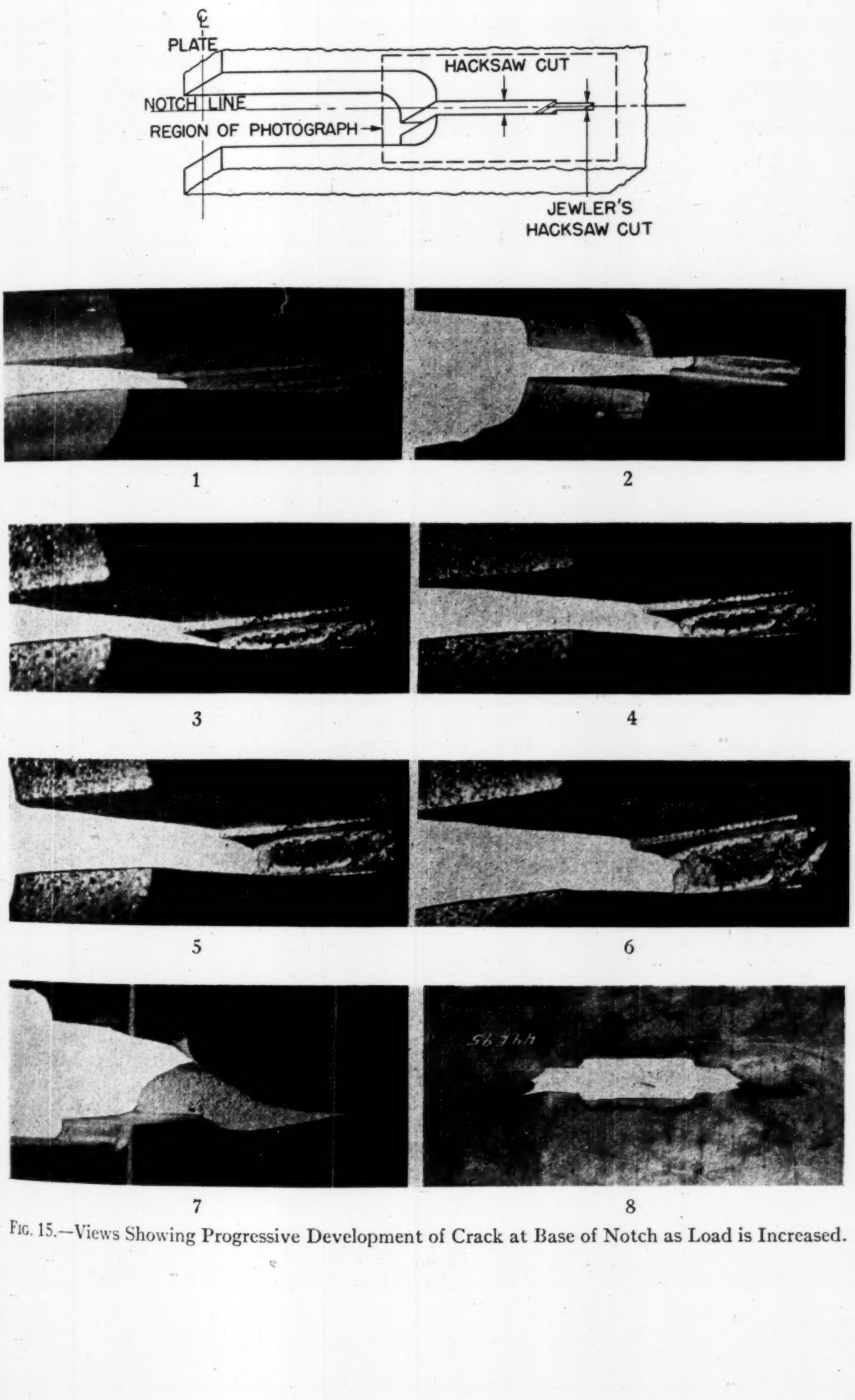


FIG. 14.—Transition Temperatures for Various Types of Specimens in Relation to Strength Properties of Steel A.

T1 = Transition temperature for simple tension bars.
T2 = Transition temperature for Charpy keyhole notch specimens.
T3 = Transition temperature for 12-in. centrally notched specimens.
T4 = Transition temperature for 3-in. edge notched specimens.

Below about −250 F., as the temperature decreases, the fracture strength *decreases*; at or somewhat below −300 F., depending on the particular steel, the fracture strength becomes equal to the yield strength and rupture occurs entirely by cleavage with very low ductility. It is of interest to note that at the lowest test temperature steels B and C fractured at stresses somewhat *lower* than the yield strength—an observation in accord with a similar observation made by Hollomon (3).

When the state of stress is such that the maximum tensile stress is high compared with the maximum shear stress, there exists not only the possibility of causing cleavage fracture before failure by shear is imminent, but also if the difference between the fracture load and that load which is of sufficient magnitude to initiate plastic flow is small, the ductility is low. When the ratio of maximum tensile stress to maximum shear stress is low, not only may the shear stress govern the mode of failure by virtue of its magnitude, but also because the accompanying plastic flow has induced a high level of cleavage strength which cannot be reached before fracture by shear takes place. Because the cleavage strength is so markedly affected


℄ PLATE
NOTCH LINE
REGION OF PHOTOGRAPH
HACKSAW CUT
JEWLER'S HACKSAW CUT

1 2 3 4 5 6 7 8

Fig. 15.—Views Showing Progressive Development of Crack at Base of Notch as Load is Increased.

by cold working (plastic flow), as shown in Fig. 8, the state of stress may then condition the mode of fracture through its influence on ductility.

In the simple tension test at −300 F., with a ratio of maximum tensile stress to maximum shear stress of 2, cleavage fractures occurred in these mild steels. It is of interest to report that with the same steels at −300 F., in torsion tests, in which the ratio of tensile to shear stress is 1, shear-type fractures were obtained. On the other hand, if the ratio of maximum tensile to shear stress is made greater than 2 by some device such as a notch, cleavage fractures occur at temperatures higher than −300 F., depending upon the severity of the notch, as indicated by Figs. 9 to 14. In passing, it should be observed that in relation to problems involving failure, a notch acts not only as a stress-raiser, but also it may induce a ratio of tensile to shear stress of sufficient magnitude to bring the possibility of brittle cleavage fractures within the range of normal atmospheric temperatures.

Figure 14 is a diagram in which attempt has been made to summarize these concepts of the influence of stress, strain, and temperature on failure, partly by use of the data from tests of steel A. Also shown on the figure are the transition temperatures for the several types of specimens used in this study. In this case the transition temperature is taken as the temperature corresponding to which 50 per cent of the fracture surface is in the cleavage mode. The way in which cleavage strength varies with temperature is conjectural; in Fig. 14, it is indicated to be constant, although some investigators are of the opinion that it decreases slightly with increasing temperature, but at a rate presumably less than the rate of decrease of shear-fracture strength with temperature.

A complicating factor in attempting to predict quantitatively the conditions for cleavage failure is the alteration in cleavage strength by prior plastic flow; not only does prestraining in tension raise the cleavage strength, but, as has been pointed out recently by Hollomon (3), the cleavage strength is lowered by prior plastic strain in compression. This phenomenon does suggest, however, the possibility of shifting the transition temperature for a steel shape having a given notch geometry by means of prestraining.

During the tests on notched flat plates it was observed that cracks formed at the base of a notch at loads appreciably below maximum load, and that these cracks first opened midway between the two faces of the plate. This is illustrated in Fig. 15. In the succeeding paragraphs consideration is given to the implications of this behavior.

The following approximate analysis indicates that after some plastic flow has occurred both the tensile and the shear stresses, at the apex of a notch, lying in a plane parallel with the loading axis and perpendicular to the face of the plate, are a maximum at the midthickness of the plate.

Let

ϵ_a = axial strain, in a direction parallel with the axis of loading, at the midthickness of the plate,

ϵ^1_a = axial strain at the surface of the plate,

ϵ_t = transverse strain at midthickness of plate, in a direction parallel to the line defining the apex of the notch, that is, perpendicular to the surface of the plate,

σ_a = axial (tensile) stress at midthickness of plate,

σ^1_a = axial (tensile) stress at surface of plate,

σ_t = transverse (tensile) stress at midthickness of plate,
K = the relationship between stress and strain; in the elastic range, $K = E$, Young's modulus, and
μ = Poisson's ratio = say 0.3 in the elastic range, and increasing to 0.5 in the plastic range.

It was observed that during loading, transverse elements of the notch surface remained essentially straight and parallel, that is, all *longitudinal* fibers at the apex of the notch were forced to undergo equal strains. This was also checked by an experiment on a thick bar having a notch of sufficient size so that SR-4 strain gages of $\frac{1}{8}$-in. gage length could be placed along the apex of the notch. This is a basic assumption in the analysis.

Since longitudinal fibers at the surface of the plate are free to contract laterally,

$$\epsilon'_a = \frac{\sigma'_a}{K} \quad \ldots\ldots\ldots\ldots\ldots (1)$$

Since longitudinal fibers at midthickness of plate are not free to contract laterally, a transverse stress, σ_t, is developed and

$$\epsilon_a = \frac{\sigma_a - \mu\sigma_t}{K} \quad \ldots\ldots\ldots\ldots (2)$$

From the assumption that $\epsilon_a = \epsilon^1{}_a$, and for conditions of elastic strain so that $K = E$ = constant,

$$\sigma'_a = \sigma_a - 0.3\sigma_t \ldots\ldots\ldots\ldots (3)$$

Equation 3 also defines the relationship which would exist after removing the load from a specimen which had been strained into the plastic range. Similarly, the general relations between stresses and strains in the transverse direction are given by

$$\epsilon_t = \frac{\sigma_t - \mu\sigma_a}{K} \quad \ldots\ldots\ldots\ldots (4)$$

Measurements indicated that the transverse strain at midthickness of plate was practically zero. Basing upon this observation the assumption that $\epsilon_t = 0$, by use of Eq. 4

$$\sigma_t = 0.3\sigma_a \text{ in the elastic range} \ldots\ldots (5a)$$

and

$$\sigma_t = 0.5\sigma_a \text{ in the plastic range} \ldots\ldots (5b)$$

For conditions of elastic strain, substituting Eq. 5a in Eq. 3,

$$\sigma'_a = \sigma_a - 0.3(0.3\sigma_a) = 0.9\sigma_a \ldots\ldots (6)$$

that is, the axial tensile stress is greater at midthickness than at the surface of the plate. For the plastic range, these stresses cannot be evaluated exactly, but assuming that the values of K are approximately the same, so that Eq. 3 holds approximately, by use of Eq. 5b,

$$\sigma'_a \text{ approaches } \sigma_a - 0.5(0.5\sigma_a), \text{ or } 0.75\sigma_a \ldots (7)$$

Since at a surface, the maximum shear stresses are one half the maximum tensile stresses, it follows that the maximum shear stresses also occur at the midthickness of the plate.

Notched bars which fail largely by cleavage often develop initial shear fractures in the same manner as those which fail entirely by shear. In such cases, the initial shear fractures extend only for a short distance inward from the notch and usually do not extend to the surface of the plate. These initial shear-fracture areas are often visible on specimens which fail by cleavage and have a characteristic crescent or thumbnail shape which differs in texture from the remainder of the fractured section.

While the preceding discussion offers an explanation of the fact that cracks start midway between the faces of a plate, it does not explain how the transition temperature is raised by notch action.

As long as the notch surface at the apex has a radius greater than zero, the ratio of the maximum tensile to maximum shear stress should be 2, so that the transition temperature should not be greater than that given by a tension test of an unnotched bar.

The following explanation is suggested as a means of visualizing how a state of triaxial stress may be developed in the region at the apex of the notch, such that the ratio of maximum tensile to maximum shear stress may be raised above 2. Consider a flat plate containing at its edge a deep, pointed notch, so sharp that the radius of curvature at the apex is, in effect, zero. Such a condition is approached when a crack is initiated. When a distributed axial load is applied to the ends of the plate, the portions of the plate on either side of the notch, outboard from the apex of the notch, tend to act in a fashion similar to the action of cantilever beams, producing tensile stresses in a direction perpendicular to the loading axis and parallel to the faces of the plate. These stresses serve to lower the maximum shearing stresses, inhibit plastic deformation, and provide conditions favorable for cleavage fracture at a relatively high temperature.

Notched bars in which the radius of curvature at the apex of the notch is large, commonly fracture first by shear for a short distance, and then, if the temperature is in a critical range for the particular steel, fail in the cleavage mode over the remainder of the cross-section, as illustrated in Fig. 12.

Elementary considerations such as those which have been outlined also indicate the magnitude of the transverse stresses (acting in a direction perpendicular to the plane of a plate) depends upon the thickness of the plate. Since the transverse stress must be zero at each face, it would not be expected to attain a high magnitude at the midplane in a relatively thin plate. Thus, with a given notch, the transition temperature should depend upon the plate thickness, being higher for greater thickness of plate. The data shown in Fig. 13, which shows the transition temperatures for 3-in. notched bars of various thickness, substantiate this conclusion.

SUMMARY

1. The range in temperature designated as the transition-temperature range, above which a steel fails entirely by shear, below which it fails entirely by cleavage, and within which a mixture of the two modes of fracture occur, is a function of the state of stress and the variation in the properties of the steel with temperature and with plastic deformation.

2. When the state of stress is such that the ratio of maximum tensile to maximum shear stress is 2, the stress condition developed (or approximated) by a plain, unnotched, cylindrical tension bar, the transition temperature range is very low. In the tests reported herein, a temperature of about −300 F. was necessary in order to obtain a fracture entirely by cleavage. When the ratio of maximum tensile to maximum shear stress is 1, a stress condition developed by a cylindrical bar in torsion, the transition temperature would be lower than −300 F., if it should be possible to attain cleavage fractures under this state of stress.

3. When the ratio of maximum tensile to maximum shear stress in a loaded bar is increased, say by means of a discontinuity such as a notch, the transition temperature is raised. The stress conditions induced by hack-saw cuts in the edges of 3-in. wide bars of $\frac{3}{4}$-in. mild steel plate were such as to bring the

transition temperatures within the normal atmospheric range.

4. While the average stress at failure may vary with the extent to which a notch induces stress concentration, the possibility of inducing cleavage fractures at relatively high temperatures depends upon the effectiveness of the notch in suppressing the maximum shearing stresses and minimizing plastic deformation. It appears that an extremely sharp notch apex is necessary in order to promote these effects. The development of an initial crack, sometimes at a load considerably below the maximum load, rather than the original notch geometry, may be a critical factor in establishing the transition temperature.

5. The thickness of plate, through its influence upon the induced transverse stresses, affects the transition temperature of notched bars, higher transition temperatures being obtained with thicker plates.

6. The cleavage fracture strength is markedly affected by plastic deformation, so that if the initial notch geometry is such that considerable plastic deformation develops before a crack is initiated, a relatively low transition temperature may be exhibited.

Acknowledgments:

The work described in this paper was conducted at the University of California, in the Engineering materials Laboratory, of which Raymond E. Davis is the Director. The laboratory is a part of the Department of Engineering of which M. P. O'Brien is Chairman.

The studies described herein were initiated in connection with an investigation of the causes of brittle behavior of ship steels which was originally conducted under a contract with the U. S. Office of Scientific Research and Development, and subject to the general direction of the War Metallurgy Committee through a project advisory committee. The investigations were continued under a U. S. Navy Bureau of Ships[3] contract, with the advice of the Committee on Ship Construction of the Division of Engineering and Industrial Research of the National Research Council. Stimulus to research on the general problem of causes of brittle failure of mild steels was given by the Board to Investigate the Design and Methods of Construction of Welded Steel Merchant Vessels and its successor, the Ship Structure Committee.

The authors take this opportunity expecially to express their appreciation to the supervisory and liaison officers acting for these agencies for friendly cooperation and support. Thanks are also due to our co-workers and assistants on the project staff for their contributions in the collection of the data.

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[3] The opinions expressed in this paper are those of the authors and do not necessarily represent those of the Navy Department or the Ship Structure Committee.

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DISCUSSION

Mr. Howard Scott.[1]—I should like to make a few comments on the Davis, Parker, and Boodberg paper. The authors apparently attach considerable importance to the ratio of shear to tensile strength. It seems to me that this is the most significant variable in determining the effects of combined stresses. They have also distinguished very clearly between shear and tension fractures, although it is rather difficult to visualize their viewpoint as presented in such a short time without preprints.

Another point is their use of the term cleavage fracture on which I should appreciate having the authors' comment. Actually cleavage fracture was originally used to designate fracture on cleavage planes through a crystal, whereas the brittle type of fracture they are discussing is probably between grain boundaries.

Mr. A. B. Bagsar.[2]—We have been very much interested in this general problem for the past several years. Our experience has been that more illuminating results are obtainable by the use of a coupon in which eccentric instead of centric loading is present.

We know that most service failures are not caused by symmetrical or centric loading and that many of such failures are due to eccentric and tangential stresses. It appears, therefore, that if eccentrically loaded test coupons had been used, some of the basic conditions encountered in service would have been simulated and I believe many of the conclusions would have been arrived at more directly and at atmospheric temperatures. Some of the test results we have obtained show, for example, that the ratio of yield load to breaking load approaches unity if notch radii of the order of 0.001 to 0.002 in. and notch angles of 30 to 45 deg. are used, in an externally notched tension bend or "tear" test coupon of rectangular cross-section. The divergence between the yield and breaking loads becomes greater as the angle of notch or the radius at the base of the notch increases. By the use of the above type of coupon, the difference between shear fractures and cleavage fractures can be determined directly by unit breaking loads, in terms of pounds per square inch. It is also possible by this manipulation and by the selection of the proper ratio of loading eccentricity to section depth and other factors, to eliminate most of the "mass effect" of the test coupon.

Messrs. H. E. Davis, E. R. Parker, and Alexander Boodberg (authors' closure by letter).—The authors wish to thank Mr. Scott and Mr. Bagsar for their discussions of and their interest in this paper. In reply to the question raised by Mr. Scott, the brittle cleavage type of failure referred to in the text is a transcrystalline type of fracture which follows the (100) planes in the ferrite crystals. Reference 1 entitled "A Study of the Tension Test" describes cleavage fractures in considerable detail.

The eccentric loading test to which Mr. Bagsar referred is a very useful test in determining the relative transition temperatures for various steels. The work of Mr. Bagsar in this field of investigation has been very interesting and useful. It was not the purpose of the present paper, however, to advocate the use of any special test for the purpose of determining the transition temperatures of steels, but rather to discuss the problem of brittle fracture of steels in a general way and to try to correlate the behavior of simple tension specimens with various notched specimens of the same material.

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AN EXPERIMENTAL STUDY OF THE PROPAGATION OF PLASTIC DEFORMATION UNDER CONDITIONS OF LONGITUDINAL IMPACT*

By P. E. Duwez[1] and D. S. Clark[1]

Synopsis

The theory of plastic strain propagation is reviewed with reference to longitudinal impact. Special impact testing equipment is described by which data have been secured for the verification of the theory. Tests in tension on long wires and on specimens with a gage length of 8 in. are reported, together with the results of some tests made in compression. The effect of release of loading and reflection of plastic strain waves on plastic strain distribution are considered. The concept of critical velocity is discussed. The anomalous behavior of material for which there is a yield point is presented. The results indicate very satisfactory agreement between theory and experiment.

Prior to 1942, the study of the behavior of metals under dynamic conditions was concerned principally with the assembly of data from notched bar impact tests. There had been some activity toward obtaining information on the performance of metals in tension impact (**1–7**).[2] In spite of the abundance of data, there was not a clear fundamental understanding of the conditions involved in impact loading, except in the case of elastic behavior. It was well recognized that elastic strains were propagated in bars with the velocity of sound, but when plastic strain was involved the behavior of the bar could not be interpreted. If materials are to be subjected to impact loading and tested in that manner to destruction, it is necessary to interpret the results in a manner that will have significance in practical applications.

It is recognized that many machine parts and structures are subject to impact loading and also that the performance of some materials under dynamic loading is different from that observed under static conditions. To explain these differences, a fundamental concept must be established. It is the development of this fundamental concept with which workers at California Institute of Technology have been concerned in the hope that a basic understanding of the dynamic performance of materials could be obtained.

Late in 1941, Theodore von Kármán conceived a theory upon which the performance of bars subjected to longitudinal impact can be based when plastic strains are involved. A paper presenting the basic theory of plastic strain propagation was transmitted to the National Academy of Science in 1941. Because of the situation existing at that time, the paper was not published, but the National Defense Research Committee made it available on a restricted basis (8).

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Research Engineer and Associate Professor of Mechanical Engineering, respectively, California Institute of Technology, Pasadena, Calif

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 522.

Beginning in 1942, the National Defense Research Committee requested the California Institute of Technology to carry on additional work in this field under an Office of Scientific Research and Development contract.

The purpose of this paper is to review the fundamental theory of plastic strain propagation and to present experimental evidence of the validity of the theory. The material presented in this paper is only a small portion of the very extensive work that has been done in this field at California Institute of Technology. Additional results will probably be published in the future.

REVIEW OF THEORY OF PLASTIC STRAIN PROPAGATION

The basic theory and early experiments on plastic strain propagation have been presented (9). The theory in its complete form is to be given in a forthcoming publication by von Kármán and Bohnenblust.

The theory of plastic strain propagation was developed in a series of steps starting from the simplest and ideal case and progressing toward a more realistic situation. It is, therefore, most expedient to consider first the case of longitudinal impact at the end of a bar extending to infinity. From this, one may proceed to a consideration of a bar of finite length, in which the reflection of strain waves becomes important. It is also necessary to consider the effect of release of load in order to interpret the results of experimental studies.

Longitudinal Impact at the End of a Bar Extending to Infinity:

Consider a bar extending from a point $x = 0$ to infinity and assume that the end point at $x = 0$ is suddenly put in motion with a constant velocity v_1. The equation of motion of an element of the bar can be written in the form

$$\rho \frac{\partial^2 u}{\partial t^2} = \frac{d\sigma}{d\epsilon} \frac{\partial \epsilon}{\partial x} \qquad \text{............(1)}$$

in which u is the displacement of the element in the longitudinal direction, ρ the mass of the material per unit volume, t the time, σ the apparent stress (load divided by initial cross-sectional area), and ϵ the strain related to the displacement by the expression $\epsilon = \frac{\partial u}{\partial x}$. The relation between σ and ϵ cannot in general be explicitly expressed, but is given by the stress-strain curve. Equation 1 is valid only under the following simplifying assumptions: (1) the relation between σ and ϵ is not affected by the rate of strain; (2) the lateral contraction of the material, that is, the contribution of the lateral contraction to the kinetic energy, is neglected. In solving Eq. 1, it was also necessary to assume that the stress-strain curve in the plastic range is always concave downward and that the unloading takes place elastically following a straight line whose inclination is given by Young's modulus.

The solution of Eq. 1 for elastic deformation and the case of a bar of infinite length subjected to a constant velocity v_1 has been given by Young (10). This solution expresses a proportionality between the value of the elastic strain ϵ_1, which propagates along the bar, and the velocity of impact, v_1, namely,

$$v_1 = \epsilon_1 c_0$$

in which c_0 is the velocity of propagation of the elastic wave given by

$$c_0 = \sqrt{\frac{E}{\rho}}$$

where E is the modulus of elasticity.

From this equation it can be seen that when the velocity of impact exceeds the value $\epsilon_0 c_0$, in which ϵ_0 is the strain at the proportional limit, plastic deformation will take place in the bar. The more general solution of Eq. 1 gives the value of the plastic strain corresponding to a velocity of impact v_1 and a method of computing the distribution of the strain at any time along the bar. The results can be summarized as follows:

1. The plastic strain ϵ_1 near the moving end of the bar can be computed from the equation

$$v_1 = \int_0^{\epsilon_1} \sqrt{\frac{\frac{d\sigma}{d\epsilon}}{\rho}}\, d\epsilon \ldots\ldots\ldots (2)$$

Since the stress-strain curve is not explicitly given, the computation is carried out graphically. A curve of $\sqrt{\frac{d\sigma}{d\epsilon}}$ *versus* ϵ is first obtained from the stress-strain curve. The curve $\sqrt{\frac{\frac{d\sigma}{d\epsilon}}{\rho}}$ *versus* ϵ is then integrated, thus giving a relation between v_1 and ϵ_1. The strain, ϵ_1, is a function of v_1 only.

2. A plastic strain of value ϵ_n propagates with a velocity c_n given by

$$c_n = \sqrt{\frac{\left(\frac{d\sigma}{d\epsilon}\right)_{\epsilon=\epsilon_n}}{\rho}} \ldots\ldots\ldots (3)$$

in which $\left(\frac{d\sigma}{d\epsilon}\right)_{\epsilon=\epsilon_n}$ is the slope of the stress-strain curve at the point $\epsilon = \epsilon_n$. The distribution of plastic strain along the bar at a certain time t for a given velocity of impact v_1 can then be determined. Referring to Fig. 1, the elastic strain ϵ_0 reaches a point x_0 given by $x_0 = c_0 t$. The plastic strain ϵ_1 as given by Eq. 2 is constant for a length x_1, given by $x_1 = c_1 t$, c_1 being given by Eq. 3, in which $\frac{d\sigma}{d\epsilon}$ is the slope of the stress-strain curve at the point at which $\epsilon = \epsilon_1$. Any plastic strain ϵ_n between ϵ_0 and ϵ_1 travels a distance x_n, given by $x_n = c_n t$, in which c_n is given by Eq. 3.

3. There exists a velocity of impact, called the critical velocity, above which rupture will occur near the impacted end of the bar, and the remainder of the bar will be essentially free of plastic deformation. The critical velocity v_c may be computed from the equation

$$v_c = \int_0^{\epsilon_m} \sqrt{\frac{\frac{d\sigma}{d\epsilon}}{\rho}}\, d\epsilon \ldots\ldots\ldots (4)$$

in which ϵ_m is the strain at which the slope of the stress-strain curve becomes zero.

Longitudinal Impact at the End of a Bar of Finite Length:

The theory of propagation of plastic deformation has been extended to the case of a bar of finite length. Two important special cases can be treated mathematically, namely, the case in which one end of the bar is rigidly fixed, and the case in which both ends of the bar are free. The methods used in this generalized theory cannot be explained without the aid of the mathematical development. The principle, however, involves the assumption that the plastic strain waves are reflected at the end of the bar somewhat like elastic waves according to a set of rules which are essentially a generalization of those applied to elastic waves.

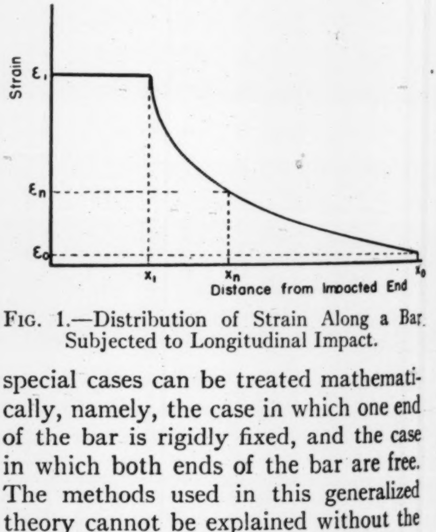

FIG. 1.—Distribution of Strain Along a Bar Subjected to Longitudinal Impact.

At the fixed end of a bar, for example, the oncoming plastic strain will be reflected as a larger strain which propagates back toward the impacted end of the bar. At the free end of a bar the stress must remain zero, and therefore the reflected wave is elastic and propagates toward the impacted end of the bar with the velocity of sound.

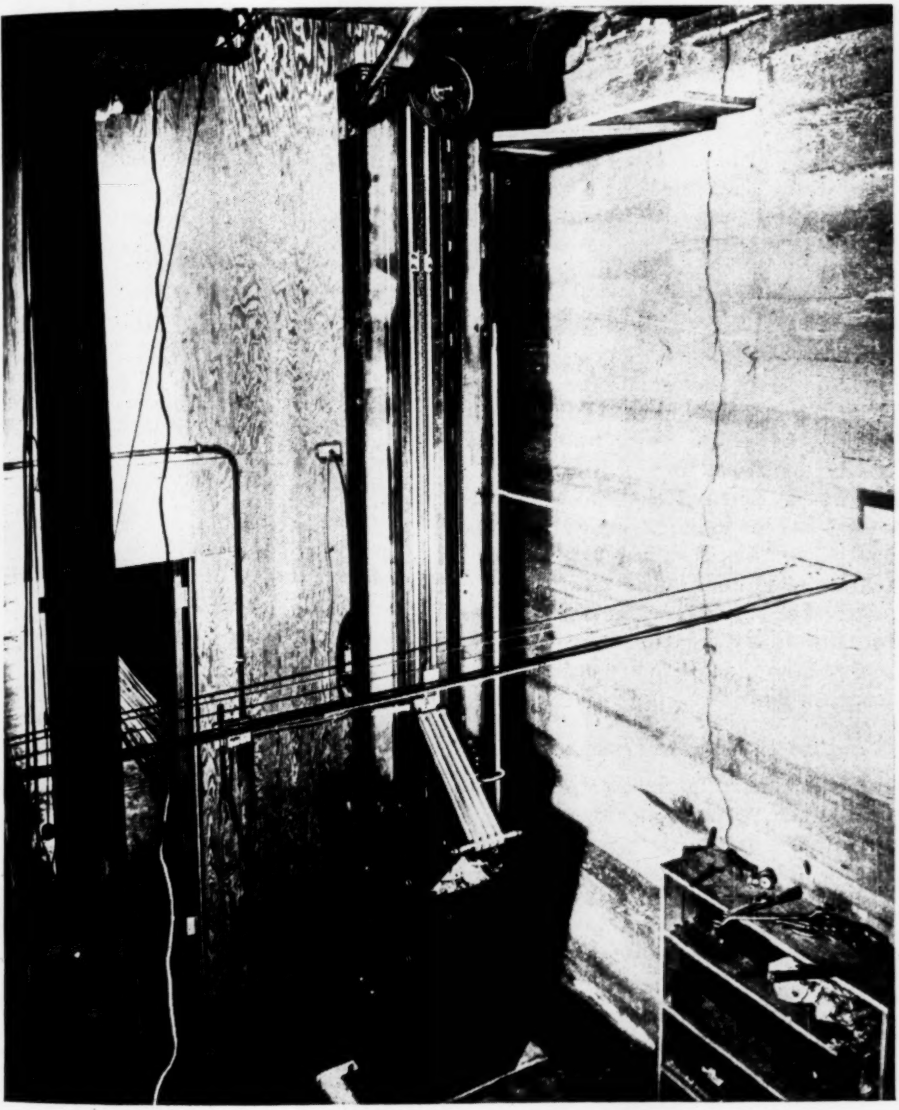

FIG. 2.—"Sling Shot" Impact Testing Machine.

Based upon these principles the strain distribution in a bar of finite length subjected to a known impact velocity can be computed. A set of curves showing the strain distribution along the bar can be constructed in which each curve corresponds to a specific time after the beginning of impact. From the stress-strain relations another set of curves showing the stress distribution can be constructed.

Utilizing the same method of analysis, the variation of stress as a function of time at any point in the bar, and in particular the stress-time relation at the fixed end of the bar, can be obtained.

EQUIPMENT FOR EXPERIMENTAL VERIFICATION OF THE THEORY

In verifying the theory of the propagation of plastic deformation, two types of testing machines have been employed, namely, a vertical "sling shot" impact testing machine, and a rotary impact testing machine. The "sling shot" machine has been used for the study of propagation of plastic strain in long wires in tension and 12-in. long rods in compression. The rotary machine has been used for the study of the propagation of plastic strain in specimens having a gage length of 8 in.

"Sling Shot" Machine-Tension:

For the investigation of the propagation of plastic strain in long wires, the machine is subject to three requirements, namely, (1) instantaneous application of a constant velocity to the free end of the wire; (2) continued motion of the free end of the wire at the impact velocity for a specified time; and (3) instantaneous release of the deforming force after the lapse of the required time. The "sling shot" machine shown in Fig. 2 satisfies these requirements. The specimen, in the form of a wire as long as 80 in. in length and approximately 0.100 in. in diameter, is held vertically in a central position between the rails of the machine. The upper end of the specimen is fixed rigidly in a grip. A tup, attached to the lower end of the specimen, is held in position by a guide tube, as shown in Fig. 3. The hammer, which weighs 3.75 lb. and slides on two vertical rails, is provided with a central hole through which the specimen passes. As many as eight rubber bands, $\frac{3}{8}$ in. thick, 1 in. wide, with an undeformed inside diameter of approximately 8 in., are attached to the hammer and fastened to the frame of the machine. The hammer is raised by a hoisting mechanism to the desired height or extension of the rubber bands. When the hammer is released from the hoisting mechanism, it is accelerated downward by the rubber bands and strikes the tup resting on the subtup of the specimen, Fig. 3. The hammer and tup move downward with a constant velocity v_1, pulling the wire until the tup

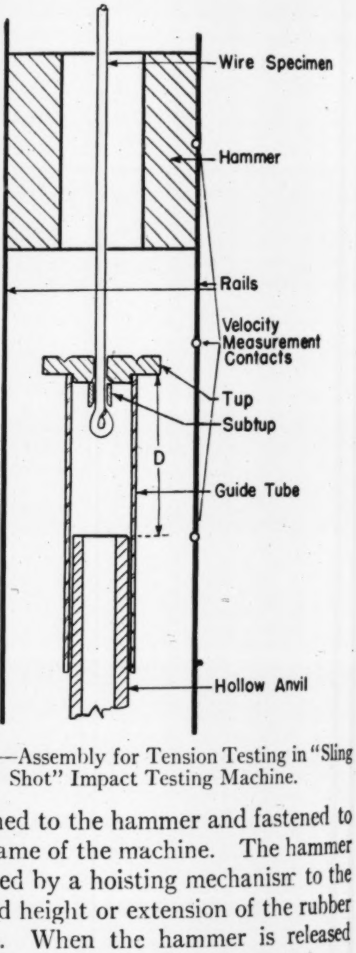

FIG. 3.—Assembly for Tension Testing in "Sling Shot" Impact Testing Machine.

reaches the anvil which has been set at such a position as to give the desired duration of impact, that is, the time during which the deforming force acts. When the hammer strikes the tup, the rubber bands are still extended, hence they continue to exert a force on the hammer as it moves toward the anvil so that its velocity is constant during this motion. When the tup reaches the anvil, some kinetic energy is stored in the wire, and this energy is converted into strain energy in the wire before the wire stops moving; therefore the subtup leaves the main tup, which has been stopped by the anvil, and continues to move downward inside the hollow anvil until the kinetic energy of the wire has been dissipated in strain energy. Upon impact of the tup with the anvil, the outer ring of the tup is sheared off, allowing the hammer to continue downward; after impact, the hammer is decelerated by means of a friction brake.

Any desired duration of impact is attained by the proper adjustment of the initial distance between the tup and the anvil (distance *D*, Fig. 3). The accuracy of measurement of this distance is ± $\frac{1}{32}$ in., giving an error of about 7 per cent for an impact distance of $\frac{1}{2}$ in., the smallest used, and ± 0.3 per cent for an impact distance of 10 in., the longest used. Since the accuracy of measurement of the hammer velocity is ± 2 per cent, the uncertainty in the duration of impact ranges from ± 9 per cent for the shortest time, to ± 2.3 per cent for the longest time used.

Because of the elasticity of the hammer and tup, the end of the wire does not attain the velocity of the hammer instantaneously. Similarly, the deforming force is not released instantaneously when the tup strikes the anvil.

The velocity of the hammer is measured by determining the time required for the hammer to travel from one to another of three fixed points spaced 3 in. apart and close to the point of impact. As the hammer passes each of these fixed points, it closes an electric circuit, causing a discontinuity in a carrier wave trace of known frequency on an oscilloscope screen. A photograph of the oscilloscope screen is taken and the number of cycles between discontinuities counted, thus giving the time required for the hammer to travel the known distance between the contact points. This system measures the impact velocity with an uncertainty of ± 2 per cent. The machine is capable of impact velocities up to approximately 250 ft. per sec.

Comparator-Ruling Machine:

In conjunction with the investigation of the propagation of plastic strain in long wires, a comparator ruling machine is employed. This machine is used for the purpose of marking the specimen at 1-in. intervals along its length with a fine scratch before testing and to measure the distance between scratches after testing.

The comparator consists of a carriage sliding on two horizontal round rods which are fixed to the base of the instrument, and a carefully made lead screw engaging a nut in the carriage. As the screw is turned, the carriage slides on the rods, and the position of the carriage is read on a dial graduated in thousandths of an inch which is attached to the lead screw. A low-power microscope and a scratching device are mounted on the carriage. The microscope is equipped with an eyepiece containing cross hairs which permit the accurate positioning of the carriage at any point along the specimen and thus the accurate measurement of the distance between scratches. The scratching device consists of a simple, accurately made mechanism on which a razor blade is mounted. The specimens to be marked or measured are held in position over the carriage on a suitable support parallel to the direction of the motion of the carriage. The comparator has an available travel of 39 in. The combined error of marking and measuring is 0.002 in. or less for any given mark.

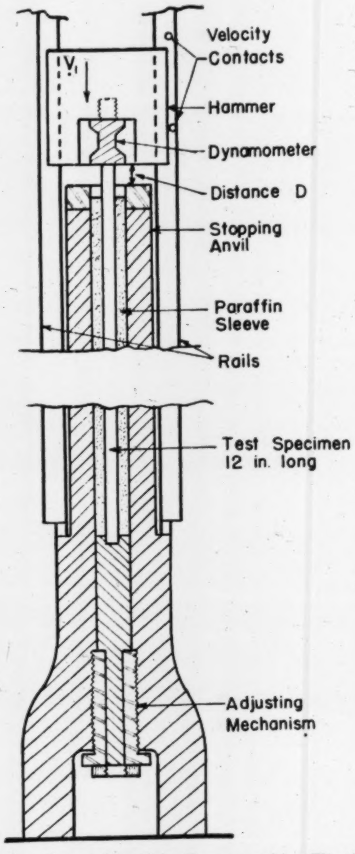

Fig. 4.—Assembly for Compression Testing in "Sling Shot" Impact Testing Machine.

"Sling Shot" Machine—Compression:

For the investigation of the propagation of plastic strain in compression, the "sling shot" machine is modified so that cylindrical specimens can be tested in compression. For these tests a mass is mounted freely between the two rails and temporarily attached to the hammer. This mass is accelerated to the desired velocity since it is in direct contact with the hammer which for these tests may be referred to as a "pusher." At the lower end of the stroke of the "pusher," to which the rubber bands previously described are attached, the free mass separates from the "pusher," traveling down the rail to contact the upper end of the specimen. The velocity of this free mass, weighing about 7 lb., is determined in the manner previously described.

The lower portion of the machine is illustrated in Fig. 4. The specimen 12 in. long and $\frac{3}{8}$ or $\frac{1}{4}$ in. in diameter is mounted vertically within a heavy, hollow cylinder. The space between the specimen and the cylinder is filled with paraffin. The upper end of the specimen is set slightly above the top surface of the cylinder. Hence, the impacting hammer hits the specimen first and is stopped a short time later by the cylinder acting as an anvil. The distance traveled by the hammer while in contact with the specimen (distance D) is adjusted by means of a screw mechanism. The distance D divided by the impact velocity determines the duration of impact.

In some tests, the force acting on the upper end of the specimen during impact is measured by means of a dynamometer fastened to the hammer. The dynamometer consists of a steel cylinder to which is cemented an electric resistance sensitive strain gage. A record of the force acting on the dynamometer as a function of time is obtained on the screen of a cathode ray oscilloscope.

Rotary Machine:

The rotary impact testing machine consists of a 750-h.p. hydraulic impulse turbine with an exciter generator. The water-wheel buckets have been removed, and the machine, with suitable electric wiring for operation as either a variable speed motor or a generator, is installed in a pit below the floor level (Fig. 5). To the wheel, which is 44 in. in diameter, are attached a pair of striking jaws and counter weights. The wheel turns on a horizontal shaft, and together with the shaft and rotor weighs approximately 2000 lb. In operation, the wheel is brought up to the desired speed, and at the proper instant a yoke is moved up into the path of the jaws by means of a strong spring which is released by a solenoid operated trigger. The jaws engage the yoke, which, in turn, strikes a tup fastened to the specimen, thus producing the impact. Figure 6 shows the specimen, tup, yoke, and jaws in position before impact. To measure the impact velocity, a strobotac is used in conjunction with appropriate strips of paper fastened to the sides of the rotating wheel. The peripheral velocity of the jaws is determined from the speed of the wheel and the distance from the center of rotation to the point of impact.

The uncertainty in the velocity measurement is ± 2 per cent or less. The machine is capable of impact velocities ranging from about 20 to 200 ft. per sec. The electrical connections to the machine are arranged so that the wheel can be stopped from any operating speed within approximately 20 sec. by operating the machine as a generator and dissipating the electrical energy through a suitable resistance.

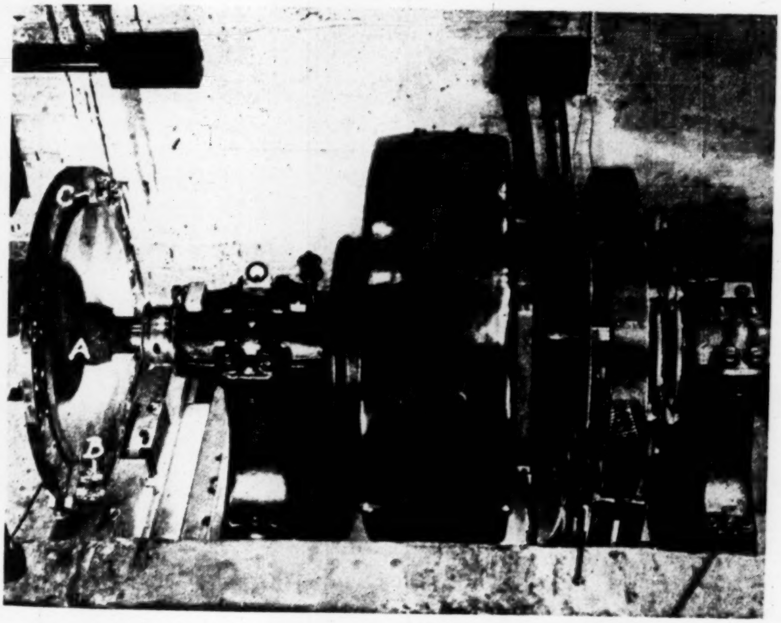

FIG. 5.—Rotary Impact Testing Machine.

The force-measuring unit, or dynamometer, into which the specimen screws, is firmly attached to one end of a 715-lb. anvil bolted rigidly to the base of the machine. The force acting at the fixed end of the specimen is determined as a function of time by means of the dynamometer. The dynamometer is made of heat-treated S.A.E. 4130 steel, on which is cemented a resistance sensitive strain gage. The electric circuit is so arranged that a change of resistance produced by a force acting on the dynamometer causes a change of voltage in the circuit. This voltage change is amplified and produces a vertical deflection on a cathode ray oscilloscope. The scale of the vertical deflection of the cathode ray beam in terms of resistance change is determined by suddenly changing the resistance of the strain gage circuit by a known amount that corresponds to a known force acting upon the strain gage as determined by a static calibration. The circuit of the oscilloscope is arranged so that a single horizontal sweep of the electron beam can be obtained at any desired speed within limits. The traces of calibration, timing line, and force-time curve on the screen of the oscilloscope are recorded photographically on 35-mm. film, with a Zeiss biotar F:1.4 lens. The uncertainty of the force measurement is ± 5 per cent or less.

Static stress-strain curves for the 8-in. gage length specimens were obtained with a 30,000-lb. Riehle universal testing machine having a least reading of 10 lb. The strain measurements were obtained with two dial gages mounted on opposite sides of the specimen. These gages measured the total strain in the 8-in. gage length. In addition to the dial gages, two Huggenberger extensometers were mounted on the specimens over a 20-mm. gage length. These gages permit a more accurate measurement of the strain for the first portion of the stress-strain curve up to a deformation of 0.6 per cent. The static stress-strain curves for the wire specimens were determined with a 3000-lb. Olsen tension-compression testing machine.

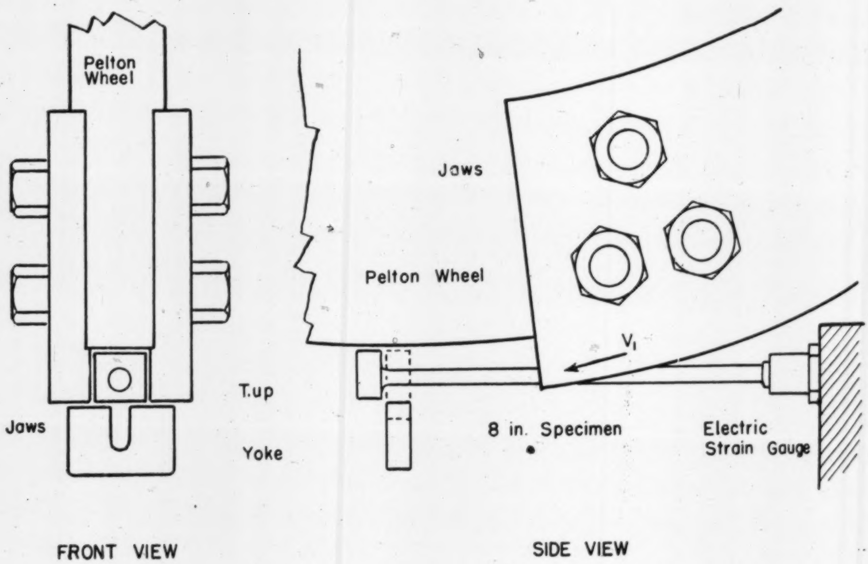

FIG. 6.—Specimen Mount and Accessories, Rotary Impact Testing Machine.

EXPERIMENTAL VERIFICATION OF THE THEORY

Study of the Distribution of Plastic Strain Near the Impacted End:

The first set of experiments on the propagation of plastic strain in tension was made in order to establish the influence of both the velocity of impact and the duration of impact on the plastic strain distribution. The specimens used in these experiments were annealed copper wires about 80 in. in length and 0.071 in. in diameter. Static stress-strain curves were obtained on two of the specimens. From the average curve the value of the slope $\frac{d\sigma}{d\epsilon}$ was first computed as a function of the strain ϵ. Using these values and applying Eq. 3, the velocity of propagation c corresponding to each strain ϵ was computed. A curve showing the relation between the strain ϵ and its corresponding velocity of propagation is shown in Fig. 7. In this particular case of annealed copper, the velocity of propagation of the elastic strain is approximately 12,500 ft. per sec. When the strain is plastic, however, the velocity of propagation decreases very markedly and is of the order of only 1800 ft. per sec. for a strain of 0.5 per cent. A strain in the range of 4 to 8 per cent propagates with a velocity of less than 1000 ft. per sec. The curve of Fig. 7 shows that the velocity of propagation falls abruptly to zero at a strain of about 16 per cent. This strain corresponds to the point of the stress-strain curve at which the tangent is horizontal.

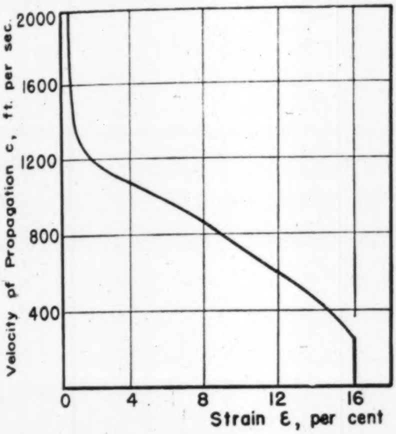

FIG. 7.—Velocity of Propagation *versus* Strain for Annealed Copper (Theoretical).

The value of the uniform strain ϵ_1 corresponding to a given velocity of impact v_1 may be computed from Eq. 2. For the case of the copper wire used in these experiments, the curve showing the velocity of impact v_1 *versus* uniform strain ϵ_1 is presented in Fig. 8. This curve terminates at a point corresponding to a strain of 16 per cent and an impact velocity of 150 ft. per sec., which is the critical velocity for this material.

As mentioned in the discussion of the equipment, marks are made on the specimen at intervals of 1 in. The distance between the origin (impacted end of the wire) and each mark is measured before and after the test. If x_n denotes the distance from the origin to a mark n before the test, and if x_n' is the distance between the two points after the test, the difference $x_n' - x_n$ represents the total

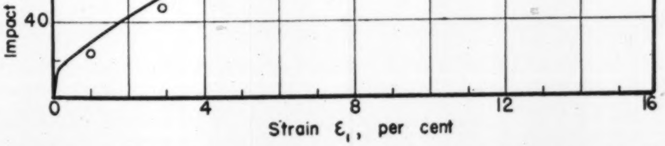

FIG. 8.—Velocity of Impact *versus* Uniform Strain for Annealed Copper in Tension.

elongation of the specimen between these two points. The values of $x_n' - x_n$ are then plotted *versus* x. The slope at any point of the curve so obtained gives the value of the permanent strain at that point. This way of measuring the strain has been found to be most practical. In the tests with copper wires it was not necessary to take into consideration the elastic recovery which was relatively small.

The first series of tests was made to establish that the value of the uniform strain ϵ_1 near the impacted end of the specimen depends only upon the velocity of impact. This strain remains constant while the elastic front and the plastic front travel along the specimen. In these particular experiments, the velocity of impact was always 92.50 ft. per sec., but the duration of the impact was varied. The curves in Fig. 9 give the distribution of the strain along the specimen. They indicate clearly that for a certain distance from the impacted end along the specimen the plastic strain is constant as predicted from the theory.

In a second series of tests, a series of impact velocities was employed. The total elongation was not necessarily the same for all the tests, but the stopping device was adjusted in such a manner that during the impact the plastic front traveled a distance of between 10 and 15 in. Figure 10 gives the distribution of the strain along the specimen for several impact velocities. For comparison with the theory, the experimental values of the uniform strain ϵ_1 are plotted as points in Fig. 8. The solid curve in Fig. 8 represents the theoretical relation between uniform strain and impact velocity. The agreement between the experimental results and the computed curve is fairly good.

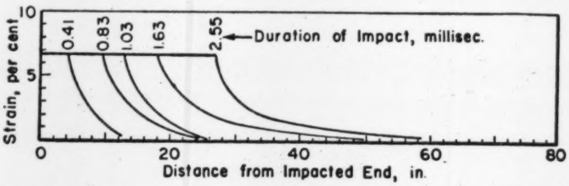

FIG. 9.—Strain Distribution for Annealed Copper in Tension. Impact velocity 92.5 ft. per second.

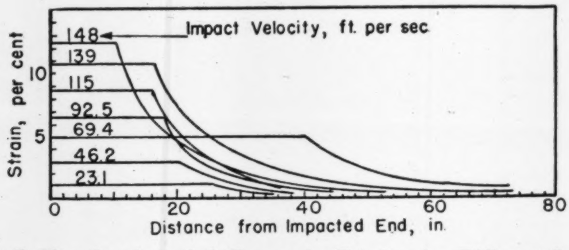

FIG. 10.—Strain Distribution for Annealed Copper in Tension for Different Impact Velocities.

It is to be noted that at an impact velocity of 171 ft. per sec. the specimen broke within the first inch. This indicates that such a velocity is above the critical velocity, as predicted by the theory. It must be pointed out that a considerable reduction of area was observed, which indicates that the rupture was not brittle. In addition, a plastic strain of relatively small intensity (less than 2 or 3 per cent) was observed along

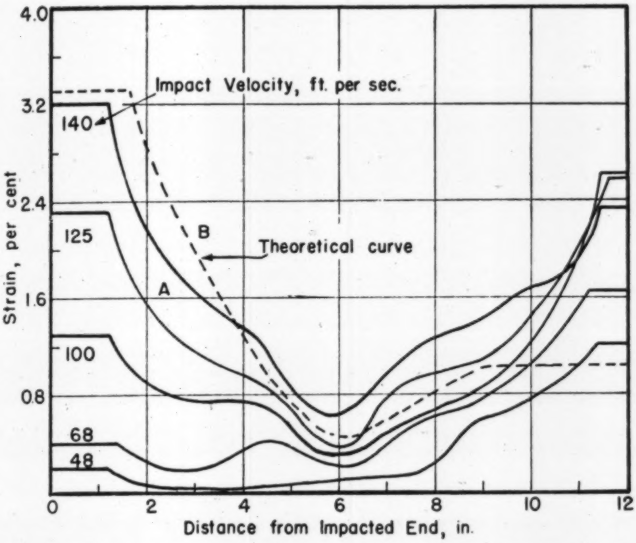

FIG. 11.—Strain Distribution for Cold-Rolled Mild Steel in Compression for Different Impact Velocities.

the wire over a distance of about 20 in. from the point of rupture. It is probable that a plastic wave started to propagate along the specimen during the time required to produce rupture.

The study of the plastic strain distribution has also been carried out in compression impact tests by the technique described in the discussion of the equipment. The theoretical computations are based on a static compression test made on cylindrical specimens having a ratio of length to diameter equal to 2. The diameter of the static test specimens was the same as that used in the dynamic tests. The compression stress-strain diagrams are obtained in several steps. The length of the specimen is measured with a micrometer and then the specimen is compressed with a particular load, unloaded, and the length measured again. This procedure is repeated for successively higher loads. The apparent stress is obtained by dividing the load by the initial cross-sectional area. From these data an apparent stress-plastic strain curve is plotted from which the elastic limit is determined. The elastic recovery is then added to each permanent strain to obtain the total strain, and thus the stress-strain diagram is constructed.

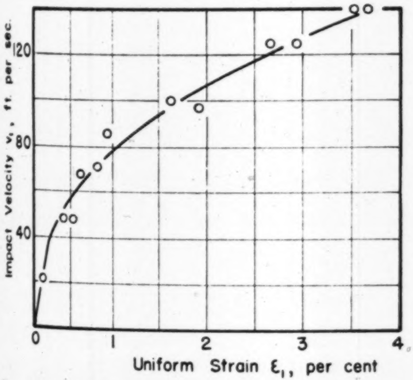

FIG. 12.—Velocity of Impact *versus* Uniform Strain for Cold-Rolled Mild Steel in Compression.

A typical series of strain-distribution curves at different impact velocities is presented in Fig. 11 for cold-rolled mild steel. These curves are essentially of a shape similar to those previously observed for copper in tension. Near the end of the specimen, which is struck by the hammer (impacted end), the strain is constant over a certain distance; the strain decreases progressively and increases near the fixed end of the specimen, because of the reflection of strain. The value of the uniform strain ϵ_1, near the impacted end, depends on the impact velocity v_1 and can be predicted by the theory. The theoretical relation between ϵ_1 and v_1 is presented for cold-rolled steel in Fig. 12, together with the experimental results. The agreement between theoretical and observed values of strain is very good. The small discrepancy noted previously in the case of the tests in tension (Fig. 8) does not seem to be present in the results of the compression tests.

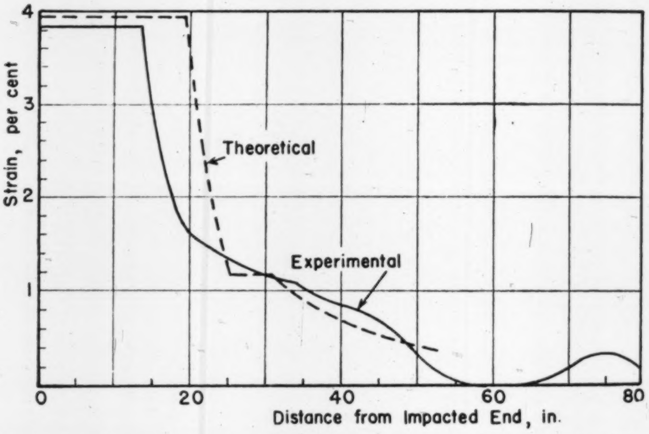

FIG. 13.—Strain Distribution for Annealed Copper in Tension. Impact velocity 62.5 ft. per second. Duration of impact, 1.3 milliseconds.

Study of the Influence of Release of Loading:

The distribution of plastic strain along a wire, as measured in the experiments described above, cannot logically be compared with the theoretical strain distribution prevailing at the instant the impact is stopped. When the action of the hammer pulling the end of the bar at a constant velocity ceases, the portion of the specimen that is in motion is not stopped instantaneously. During the deceleration period, the kinetic energy stored in the specimen is transformed into strain energy, and the total extension is larger than the distance through which the hammer has been in contact with the specimen.

The shape of the plastic wave is greatly influenced by this "stopping effect" and will be at some variance with the theoretical curve that represents the shape of the wave at the moment the impacting force is released. Numerous tests were performed in order to investigate the stopping effect and its influence on the shape of the strain distribution curve. A typical experimental strain distribution curve is shown in Fig. 13. This curve, like many others which are the result of accurate measurements, exhibits definite irregularities which are not revealed in the less accurate tests discussed in the previous section. The strain-distribution curve computed by means of the theory, taking into account the stopping effect, is also shown in Fig. 13.

The irregularities observed in the experimental curve can be related to the horizontal steps in the theoretical curve. These irregularities are not exactly steps but rather changes in curvature. The steps result from the action of the elastic stopping wave upon the plastic wave. It is probable that the complexity of the physical process of plastic slip in the metal has a tendency to smooth out this effect, and therefore the observance of definite steps is doubtful even if the accuracy of measurements was improved. Furthermore, the release of the load at the end of the test is not instantaneous. Therefore, the front of the stopping wave is not square, as assumed in the theory.

In additional tests the irregularities in the experimental curves and the steps in the corresponding theoretical curves do not always occur at the same values of x. The position of a step on the theoretical curve is determined by the point at which the stopping wave meets the plastic wave, which in turn depends on the time at which the stopping wave begins. This time (which is the duration of impact) is difficult to measure very accurately, and this may account for the observed discrepancies between experimental and theoretical results. In spite of the fact that the theory assumes that the impact is stopped instantaneously and it is recognized that in the experiment this condition does not prevail, the general shape of the strain-distribution curves can be explained satisfactorily by an analysis based on the theory of plastic strain propagation.

Study of Reflection of Plastic Waves:

In order to investigate the reflection of plastic strain waves from the fixed end of a bar, the duration of impact must be great enough to permit a large plastic strain to travel a distance greater than the length of the bar. Instead of increasing the duration of impact in tests on specimens 80 in. long, it is more convenient to use a specimen 40 in. long and to maintain the duration of impact within the same limits as in the previous tests. For these particular experiments, annealed aluminum wires were used. A typical strain-distribution curve is shown at A in Fig. 14.

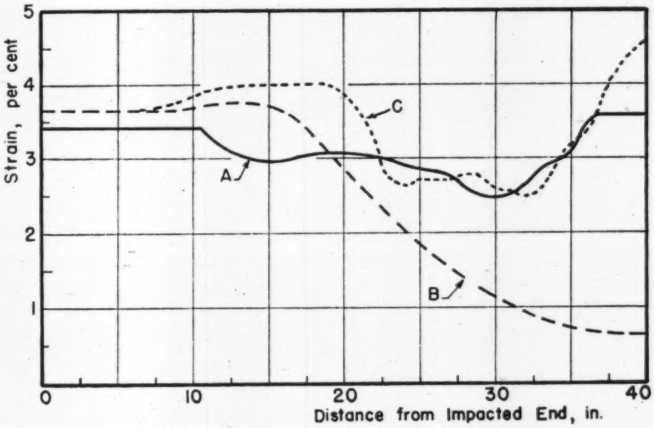

FIG. 14.—Strain Distribution for Annealed Aluminum in Tension. Impact velocity, 80 ft. per second. Duration of impact, 1.04 millisecond.

Curve A—Experimental
Curve B—Theoretical without stopping effect
Curve C—Theoretical with stopping effect

In computing a strain-distribution curve from the theory with which the experimental curves could be compared, it is recognized that if only the reflection is taken into account the agreement is not satisfactory. However, if the stopping effect is included in the analysis, the theoretical curve is a good approximation to the observed strain distribution. As an illustration, the dashed curve, *B*, of Fig. 14 is computed without considering the stopping effect, and the dotted curve, *C*, is computed by taking the stopping effect into account. It is to be noted that the theoretical curve, *B*, for which the stopping effect is neglected, is not in good agreement with the experimental curve. In particular, the computed strain at the fixed end is smaller than the measured strain. The theoretical curve, *C*, obtained by considering the stopping effect, is in much better agreement with the experimental results. The theoretical strain at the fixed end of the specimen is, in this case, greater than the measured strain. This

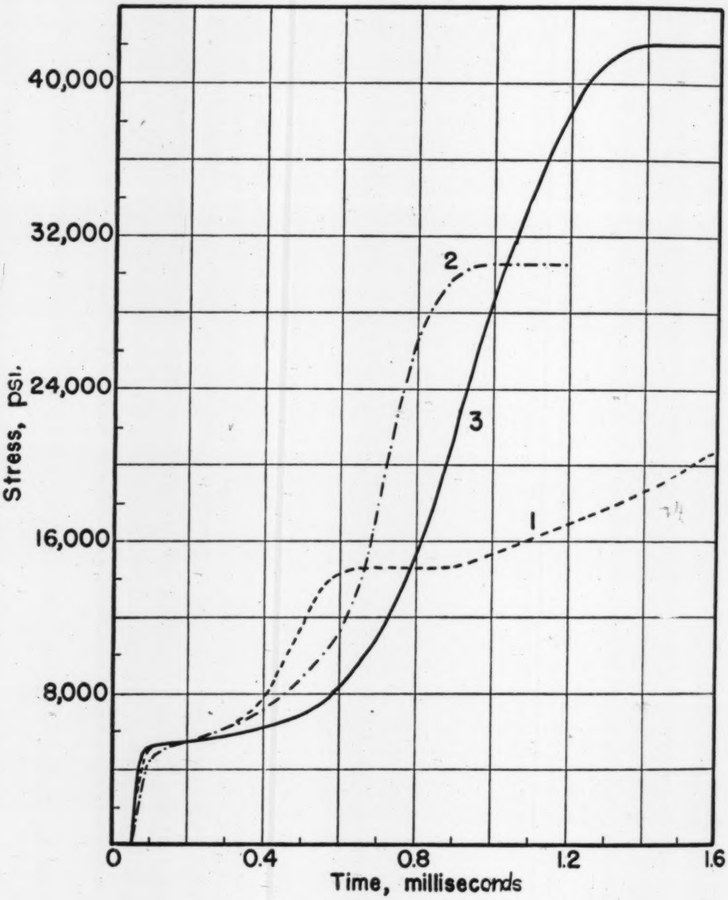

FIG. 15.—Experimental Stress-Time Curves in Tension for Fixed End of Annealed Copper. Gage length, 8 in. Impact velocities: Curve 1—25 ft. per second; Curve 2—112 ft. per second; Curve 3—200 ft. per second.

discrepancy can be explained by the fact that the reflection of the plastic strain in the experiment is not perfect, as assumed in the theory.

The effect of reflection of plastic waves from the fixed end of a specimen subjected to compression impact has also been studied. A typical example is shown in Fig. 11. Here it is to be noted that the general shape of the experimental curve, *A*, is in satisfactory agreement with the theoretical curve, *B*. In this case it is observed that the theoretical strain at the fixed end of the specimen is less than the measured strain. This result is opposite to that shown in Fig. 14 for an aluminum specimen tested in tension. However, in compression, cases have been observed in which the theoretical strain was equal to or greater than the measured strain. This behavior may be attributed to the fact that the value of the theoretical strain at the fixed end of the specimen is critically dependent upon the duration of impact which, in the case of compression, is too short to be measured accurately.

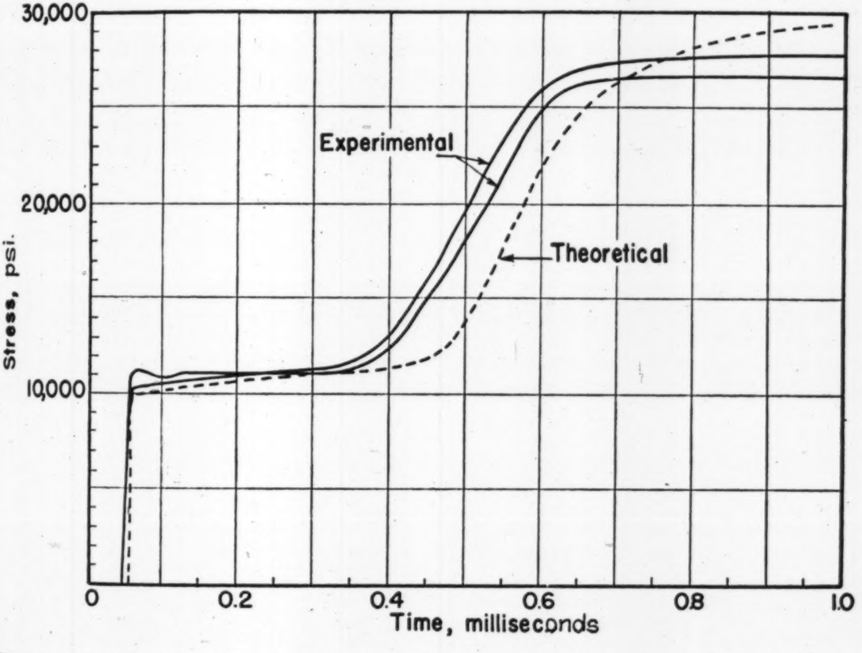

FIG. 16.—Experimental and Theoretical Stress-Time Curves in Tension for Fixed End of Annealed Copper. Gage Length, 8 in. Impact velocity, 100 ft. per second.

Force-Time Relation at the Fixed End of a Specimen:

The force acting at the fixed end of a specimen subjected to tension impact is a function of time which can be computed on the basis of the theory reviewed previously. The force at the fixed end of a specimen has been measured by making tests on annealed copper specimens with a gage length of 8 in. with the rotary impact testing machine previously described. The force-time diagrams obtained in tests made at impact velocities of 25, 112, and 200 ft. per sec. are reproduced in Fig. 15.

The shape of the force-time diagrams can be explained qualitatively on the basis of successive reflection of strain waves. From the instant of impact ($t = 0$) to a time $t_0 = \frac{l}{c_0}$, which is the time required for the elastic strain to travel the length, l, of the specimen at velocity c_0 the force at the fixed end remains zero. At the time t_0, there is a sudden increase of the force associated with the sum of the elastic strain and the reflected plastic strain. After the time t_0, the force increases gradually until the large plastic strain reaches the fixed end. Then the force increases more rapidly. This increase may be followed by an interval during which the force may remain almost constant. The magnitude of the sudden increase of stress resulting from the reflection of large plastic strain is greater the higher the impact velocity, as indicated in curves 1, 2, and 3 of Fig. 15.

A comparison between experimental and theoretical stress-time diagrams is shown in Fig. 16. The copper specimens used to secure the results reproduced in Fig. 16 have an elastic limit higher than that of the specimen for which the curves of Fig. 15 were obtained. This is shown clearly by the first sudden increase in stress at the time $t = \frac{l}{c_0}$. The agreement between theoretical and experimental curves is very satisfactory as far as their shape is concerned. The experimental curves, however, lie to the left of the theoretical curve. This discrepancy could be explained by assuming that any strain has a velocity of propagation slightly greater than that based upon the static stress-strain curve.

Force-time diagrams recorded in impact testing have been interpreted by some investigators as dynamic stress-strain curves. The transformation of the time axis into a strain axis infers that the strain is uniform all along the specimen at any instant during impact. The theory of strain propagation shows that for impact velocities greater than approximately 10 ft. per sec., this hypothesis is not justified, and a stress-strain relation cannot be determined in a high velocity tension-impact test. Any attempt to deduce an elastic limit from a force-time diagram at the fixed end is also futile, since the first sudden increase in force recorded at the fixed end of a specimen results from the superposition of the elastic wave and the reflected plastic wave. Practically, the problem is still complicated by the propagation of elastic waves in the force measuring device. This secondary phenomenon should be taken into account in interpreting force-time diagrams in high velocity impact tests.

It is important to recognize that the strain rate in impact tests varies from point to point along the specimen and for a given point is also dependent upon time. Computations based on the theory of wave propagation have shown that the average strain rate calculated on the basis of uniform strain is far from the actual strain rate even when the velocity of impact is as low as 10 ft. per sec. It is, therefore, not logical to utilize tension-impact tests to study the influence of rate of strain on the properties of metals.

SPECIAL CASE OF A METAL HAVING A YIELD POINT

The theory of propagation of plastic strain is, in its present state, applicable only to solids for which the plastic range of the stress-strain curve presents a continuously decreasing slope. This condition predominates in non-ferrous metals in the annealed or cold-worked condition, and in cold-rolled steel and heat-treated steel. For annealed iron and annealed carbon steels, the presence of a definite yield point in the stress-strain curve leads to a multivalued function for the velocity of strain propagation. In view of this condition, it is reasonable to expect that the shape of the plastic wave will be markedly different from the wave shape in a material devoid of a definite yield point.

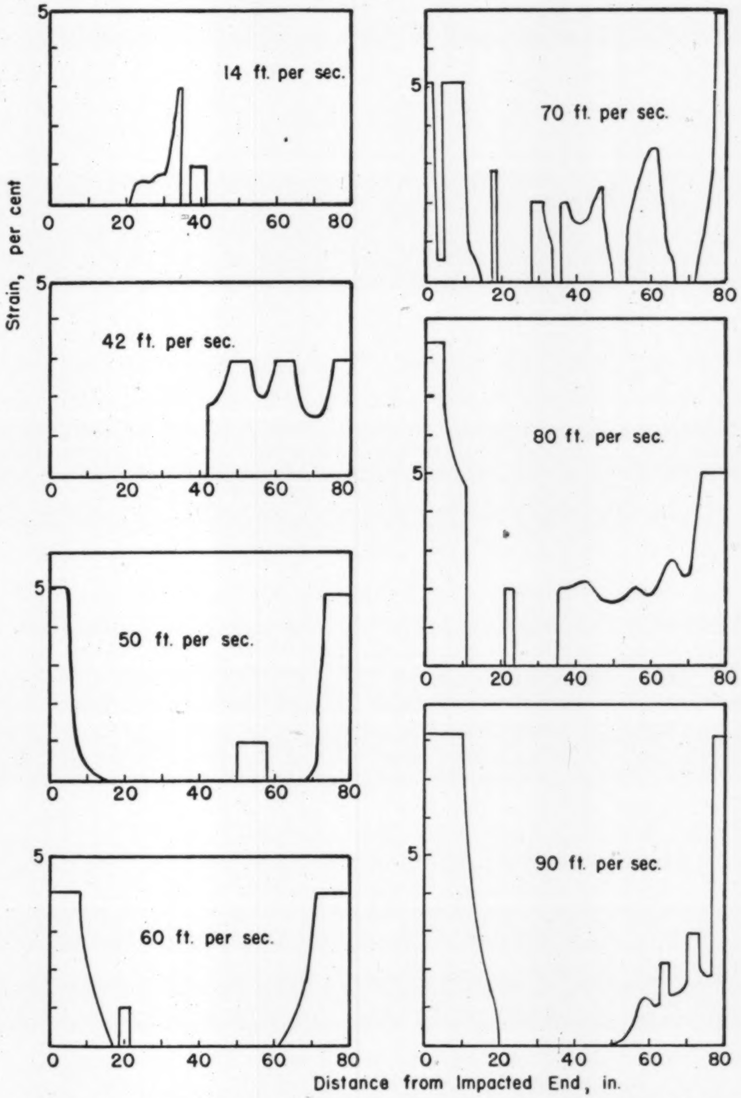

FIG. 17.—Strain Distribution for Annealed Iron Wire in Tension for Different Impact Velocities

Experiments have been made to determine the shape of the plastic strain wave in annealed low-carbon steel, in tension and in compression, at velocities up to 150 ft. per sec. The tension specimens are in the form of wires 0.12 in. in diameter and 80 in. long. The yield point is 24,600 psi.; the ultimate strength is 42,800 psi. The compression specimens are 12 in. long and $\frac{1}{4}$ in. in diameter.

The strain-distribution curve for the annealed low-carbon steel at any impact velocity differs from those shown previously for copper. The plastic strain along the specimen does not change progressively, but some portions of the specimen exhibit a plastic strain of several per cent, and each portion is separated from the others by regions in which no plastic strain can be detected. A series of strain-distribution curves in tension is shown in Fig. 17 for different impact velocities.

The most striking result of the tension-impact tests is that up to an impact velocity of 42 ft. per sec. no plastic strain occurs near the moving end, and the strain resulting from the impact is purely elastic. In this case, the equation $v_1 = \epsilon_1 c_0$ can be applied. If v_1 is 42 ft. per sec., the value of ϵ_1 is 0.25 per cent, which corresponds to a stress of 72,000 psi., or 2.9 times the static yield stress. It is, therefore, apparent that under impact conditions an elastic stress several times larger than the static elastic limit can be propagated in a metal such as iron or mild steel. It is very probable, however, that the metal could not stay indefinitely under such a state of stress after the passage of the elastic front, and plastic deformation would eventually occur. According to the present results, a stress higher than the static yield point can be sustained for at least 2 or 3 milliseconds after the passage of the wave. There is no information to indicate the order of magnitude of the time such a high stress could be sustained by the material before plastic deformation occurs.

The results of tests made with impact velocities above 42 ft. per sec. indicate the existence of plastic strain at the impacted end of the specimen. The values of the uniform strain ϵ_1, near the impacted end, increase as the impact velocity increases to about 100 ft. per sec. Above 100 ft. per sec., all the specimens tested failed within a distance of $\frac{1}{2}$ in. from the impacted end, showing that the critical velocity was reached.

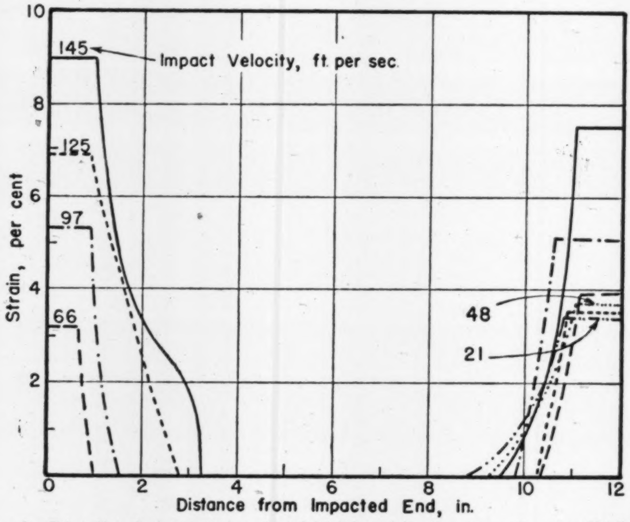

FIG. 18.—Strain Distribution for Annealed Mild Steel in Compression for Different Impact Velocities.

The strain-distribution curves shown in Fig. 17 are very inconsistent. The random character of the plastic strain measured in the central portion of the wire may be caused by local imperfections in the specimen, such as variation in the diameter of the wire, impurities, and nonuniformity in grain size of the metal. The strain distribution observed in some specimens was more nearly in accordance with that expected on the basis of the strain-propagation theory. In these instances the strain distribution seems to be the result of a propagation of a plastic strain wave with a very steep front and its reflection from the fixed end. If this explanation is correct, the velocity of propagation of the plastic strain may be computed approximately from the distance traveled by the plastic strain front and the duration of impact. The velocity of propagation has been computed from the results of four tension tests at different impact velocities. While the uniform strain ϵ_1 increases with impact velocity, the velocity of propagation of plastic strain was about the same in all cases, namely, 420, 420, 380, and 330 ft. per sec. This would seem to indicate that the velocity of propagation of plastic strain is small and not markedly dependent on the value of the strain. This differs from what has been found on impact tests with metals which do not have a yield point.

A similar series of tests was made in compression on specimens of annealed mild steel. These specimens were $\frac{1}{4}$ in in diameter and 12 in. long. The proportional limit of this material determined in a static compression test is 42,000 psi. The strain-distribution curve shown in Fig. 18 exhibits the same discontinuous character as those obtained from tension tests. (Compare Fig. 17.) As in the case of tension, the plastic strain is constant for a certain distance near the impacted end of the specimen and then drops to zero strain rather rapidly. The strain at the fixed end of the specimen in Fig. 18 results from the reflection of plastic strain. Contrary to the results obtained in the tension tests on mild steel (compare Fig. 17), isolated sections in the middle of the specimen are not plastically deformed. It has also been found that no measurable plastic strain could be detected at the impacted end for impact velocities below 50 ft. per sec. This result means that for impact velocities less than 50 ft. per sec. the strain wave originating at the impacted end is purely elastic. Following the same reasoning used in discussing the results of tension tests, it can be concluded that the elastic limit under impact conditions is at least 90,000 psi., or more than twice the value measured in a static compression test. From the data on which the curves of Fig. 18 are based, the velocity of propagation of a uniform strain ϵ_1 can be computed and is found to be equal to approximately 800 ft. per sec. This indicates that in compression as well as in tension on materials having a yield point, the velocity of propagation of plastic strain is relatively small and is apparently independent of the value of the strain. This conclusion implies that the stress-strain relation prevailing in the dynamic process may consist of two straight lines. The first straight line would join the origin to the elastic limit (the dynamic elastic limit would be two or three times the static elastic limit) with a slope equal to Young's modulus. The second straight line would have a much smaller slope from which the velocity of plastic strain propagation would be computed.

Since the theory of propagation of plastic strain in its present state does not cover the case of a material having a yield point, Eq. 4 cannot be used for the computation of a critical velocity. However, it has been demonstrated by experiment that, for example, an annealed S.A.E. 1020 steel has a critical velocity of about 100 ft. per sec. Further information on experimental work regarding the critical velocities of several materials has been secured and will be published at a later date.

CONCLUSIONS

The results of these studies show that the phenomena observed in longitudinal impact are explained by the von Kármán theory of plastic strain propagation. The agreement between theory and experiment is unusually satisfactory in spite of the approximation on which the theory is based and the difficulties of making the experiments conform to these requirements.

A clear concept of a critical velocity in tension impact is an important result of the theory of plastic strain propagation. The existence of such a critical velocity has been proven by the experimental study.

It has been shown in this discussion that the theory in its present form only applies to materials for which the stress-strain curve has a continuously decreasing slope in the plastic range. Thus, when a material having a yield point is subjected to longitudinal impact, its behavior cannot be predicted. Experimental evidence indicates, however, that the relation between stress and strain under impact conditions differs essentially from that under static conditions.

Acknowledgment:

The investigations presented in this paper were conducted at California Institute of Technology under a contract with the National Defense Research Committee of the Office of Scientific Research and Development. The authors wish to acknowledge the assistance of D. S. Wood, who performed a large portion of the laboratory tests.

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DISCUSSION

Mr. L. H. Donnell.[1]—In connection with the very valuable work which von Kármán and his coworkers at the California Institute of Technology have done on plastic wave transmission, it is of interest to review some of the parallel work which has been done elsewhere. The parallel work to which I wish to refer started with a paper of mine entitled, “Longitudinal Wave Transmission and Impact,” which was published in the *Journal of Applied Mechanics* in 1930. This paper dealt chiefly with elastic wave transmission, but in one section of it a method was presented for dealing with plastic wave transmission by the superposition of waves similar to elastic waves.

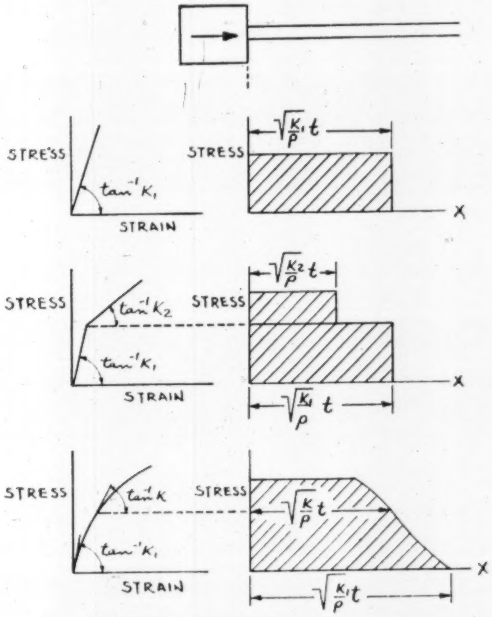
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Fig. 19.—Schematic Representation of the Principle of Superposition of Waves.

The principle of superposition of course does not apply in general to nonlinear or plastic action, but it does apply in the manner in which it was used in this method, and leads in a simple manner to results which are exact within the assumptions which all investigators in this field have made.

The accompanying Fig. 19 shows how this method is applied. The upper diagram shows an elastic wave. Such a wave is propagated along the bar with a velocity of $\sqrt{K_1/\rho}$, where K_1 is the slope of the stress-strain curve (the modulus of elasticity in this instance) and ρ is the mass density. If, as will be assumed, the stress is applied instantaneously on the end of the bar and remains constant, the wave will have at a certain time the stress distribution shown, with a square front.

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The middle diagram of Fig. 19 shows a wave in a material having a stress-strain diagram consisting of two straight lines of slopes K_1 and K_2. The stress corresponding to the lower part of the stress-strain diagram sends a wave along the rod with a velocity $\sqrt{K_1/\rho}$. The remainder of the stress sends a wave along the rod with a velocity of $\sqrt{K_2/\rho}$, the same as an elastic wave in a material of modulus K_2. This superposes on the first wave, giving a stepped wave front, as shown. The width of the step increases with time, starting with zero width when the wave front crosses the struck end at the time this force was first applied.

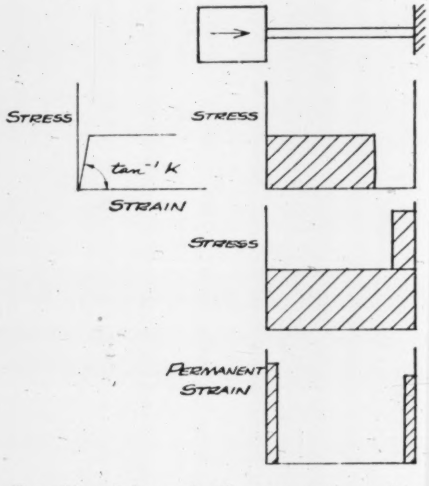

FIG. 20.—Schematic Representation of a Wave in a Material Having a Definite Yield Point.

The bottom diagram in Fig. 19 shows a wave in a material with a curved stress-strain diagram as shown. Such a stress-strain diagram can be considered to be made up of many straight sections, each of which sends a wave along the bar with a velocity corresponding to its slope. The superposition of such waves, when the number of straight sections is taken infinite, gives a wave of the type shown, with a curved sloping front whose slope increases with time.

The superposition method thus gives in a simple manner the stress-time-position relations in the bar. Since this method is based on the same assumptions, and satisfies the same physical laws as the analytical method developed independently by von Kármán in 1941, the results obtained by the two methods are identical.

Each method has its advantages and disadvantages. One advantage of the superposition method is that it readily permits an interpretation of the case of wave transmission in a material with a definite yield point, for which the analytical method gives indeterminate results. As was discussed in the paper in 1930, and as can be seen from the middle diagram, when the slope of the upper part of a two-straight-line stress-strain diagram approaches zero, the velocity of the corresponding wave also becomes zero. That is, this part of the wave *never gets started*, the energy corresponding to it going into plastic deformation at the struck end.

As shown in the top diagram of Fig. 20, the elastic lower portion of the wave moves along the bar. If the other end of the bar is fixed, the wave would, under elastic conditions, be reflected without change of sign of stress, as shown in the middle diagram. Since the superposition of the original and reflected waves would be above the yield point, the conditions at the fixed end as the wave passes it are now similar to those at the struck end as the wave started, and similar plastic flow takes place at the fixed end. The resulting residual strains will therefore be something like those shown in the lower diagram. This looks very much like the experimental results which Mr. Clark has shown for a mild steel bar when the time of impact is short enough to prevent much strain hardening.

I did not carry this method any further than the results which have been described. However, in 1941, at about the time von Kármán was developing his analytical method, Merit White and LeVan Griffis, working independently of von Kármán, started to extend the superposition method to the effects of unloading when the force ceases to act on the end of the bar, the calculation of the residual strains left in the bar, the effects of discontinuities in the bar, and the case of waves in a material all or part of whose stress-strain diagram is concave upward, for which case the purely analytical approach again gives indeterminate results. This work of White and Griffis is published in National Defense Research Committee reports and in a forthcoming paper before the Applied Mechanics Section of the American Society of Mechanical Engineers. Mr. Griffis is present and will say a few words about this work.

MR. LEVAN GRIFFIS.[2]—The accompanying Fig. 21 presents the results of the analysis of the state of plastic strain for the case of an abrupt impact on the left end of a long bar of annealed copper.

The *upper curve* shows envelope of maximum stress, and the dotted curve shows the position of the unloading elastic stress wave at the instant of overtaking the slower moving plastic wave front. The effect of the unloading upon the residual plastic strain is evident in the discontinuous steps in the maximum stress envelope, which analysis is readily handled by the superposition method.

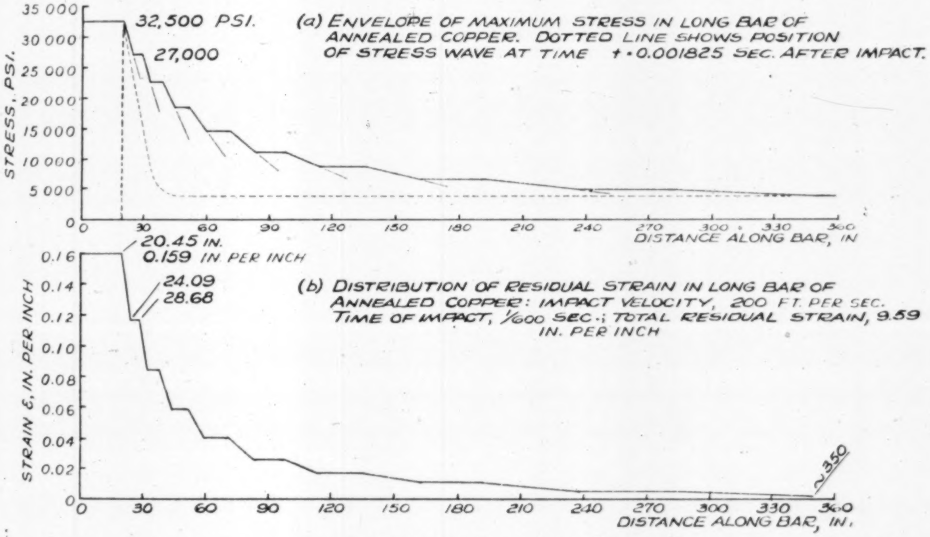

FIG. 21.—Calculated Curves Showing the Final Strained State of a Long Bar of Annealed Copper After Impact.

The *lower curve* shows the calculated residual strain. Attention is called to the initial region of maximum strain whose length is shown by analysis for sharp impact to be directly proportional to the duration of impact T: $l_1 = \frac{c_0 c_1}{c_0 - c_1} T$, where c_1 is the speed of propagation of the maximum plastic stress component.

The value of the superposition or graphical method is further shown by the ease with which it permits analysis and interpretation of the case of materials having a stress-strain curve totally or partially concave upward, as in the yield and strain hardening region of mild steel referred to by Mr. Clark. Figure 22 shows the basic method developed by M. P. White and the writer. Consider in the upper diagram the material having a stress-strain curve consisting of two straight lines. In accordance with the wave-propagation equation, the upper stress component tries to travel at a speed, $c = \sqrt{K_2/\rho}$, which is faster than the lower component. Obviously, this is not possible and the upper stress component cannot "overhang" but spills over to become part of a slower moving component as shown in the second diagram, which the upper component continues to overtake. Since the wave front cannot lean forward or backward, it propagates with a straight front, as shown in the case of the diagram for a concave upward stress-strain curve. Thus, the material behaves under impact as if it has a linear or chordal stress-strain relation. The physical interpretation of the case leads to the terminology, "shock wave," for such a wave, since it satisfies conditions similar to those discussed in mathematical physics for shock waves in fluids and supersonics.

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[2] Chairman, Department of Mechanics, Illinois Institute of Technology, Chicago, Ill.

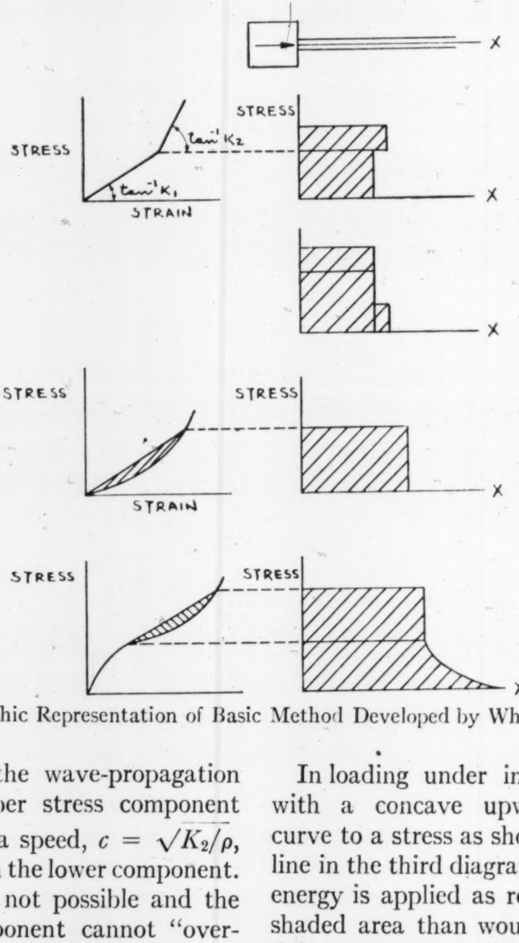

FIG. 22.—Graphic Representation of Basic Method Developed by White and Griffis.

In loading under impact a material with a concave upward stress-strain curve to a stress as shown by the dotted line in the third diagram of Fig. 22, more energy is applied as represented by the shaded area than would be required to deform the material statically to the same strain. This difference in energy is unrecoverable and is dissipated as thermal energy in the form of heat attending the impact process.

For a material with a stress-strain diagram only partly concave upward, as shown in the bottom diagram of Fig. 22, the material behaves under impact as if the stress-strain curve were chordal and partly linear. Part of the wave front is then vertical, and as shown to the right, moves faster than its lower component wants to at the point of tangency, but moves slower than its upper component tries to.

Referring now to the paper presented by Mr. Clark, it is regrettable that he has not included the characteristic curves (stress-strain curves, that is) for the materials which he studied, notably the annealed copper and annealed mild steel.

Lacking these data, the writer analyzed in accordance with procedures outlined above the case of impact on another mild steel having a definite yield region, with the following results:

For impact velocities as given, the length of the initially deformed region of maximum strain is calculated, and also the maximum strain: for $T = 0.0001$ sec.

If v = 30 ft. per sec.	l_1 = 1.8 in.	δ = 0.008 in. per in.
40 ft. per sec.	2.2 in.	0.011 in. per in.
80 ft. per sec.	3.5 in.	0.026 in. per in.

This material has a yield of 30,000 psi, corresponding to a yield impact speed of 17 ft. per sec. Since, by assuming definite yield and a duration of impact of 0.1 millisecond, the predicted strain is so small as to be very difficult to measure by the equipment described in the paper, it is questionable whether the author can conclude from his experiments that the impact elastic limit is raised by a factor of three times the static yield.

Similarly, since the length of maximum strained region is directly proportional to T, the duration of impact, it would seem that until more accurate information is available on the experimental time of impact duration, the behavior of the material cannot be definitely established as purely elastic.

Attention is called here to the author's conclusion that an elastic stress of 72,000 psi. might be maintained for as long as 2 milliseconds. This seems questionable if one remembers that in such a time interval an elastic stress will travel 400 in. in steel, and would thus be reflected five times from the ends of the 80-in. wire tested, increasing the stress at each reflection by 72,000 psi., and resulting in an "elastic" stress of 216,000 psi. at the struck end.

Finally, the conclusion that the speed of the propagation of the upper components of the plastic wave is independent of the strain intensity seems contrary to the entire basic theory, much of which theory has been well substantiated by the excellent work of Clark and his associates. It is hoped that the computations and data upon which this conclusion is based will be presented later for discussion. It is true that for impacts resulting in maximum strain values slightly above the yield, the wave speed of the maximum stress component will follow the chordal lines shown in Fig. 22, which chordal or linear curves will correspond to very slow wave speed over a fairly wide range of impact velocities. This may further substantiate the analysis discussed above.

Mr. Merit P. White[3] (*by letter*).—This interesting and valuable report of the work done at the California Institute of Technology in recent years will be welcomed by all those investigators who are concerned with the behavior of materials under dynamic conditions, and especially by those who have not had access to the classified reports of this and other work that appeared in the United States and Britain during the war. For the benefit of readers who have not had access to all the literature on this subject it might be well to mention briefly some of the theoretical investigations that have dealt with the propagation of plastic deformation under longitudinal impact. Between 1941 and 1943 most work in the United States in this field, both experimental and theoretical, was carried out under Division 2 of the National Defense Research Committee. Practically all theoretical work that has been done on this subject was completed during this period.

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[3] Scientific Consultant, War Department, Washington, D. C.

In 1930 L. H. Donnell[4] showed that in a medium having a concave downward stress-strain relation a longitudinal wave travels with continuous change of shape. Donnell gave the relation between the stress-strain characteristics of the medium, the rates of travel of elements of the wave, and the shape of the wave at any time. Late in 1941 three independent studies of wave propagation in plastic media were made. One of these was by von Kármán and is described by Duwez and Clark. This treated the case in which a constant impact stress or constant impact velocity is applied continuously to the end of a very long uniform wire. This analysis gave the relation between the impact velocity and the resulting stress and strain distribution along a wire at any instant and defined the critical impact velocity at which more or less instantaneous rupture would occur at the point of impact. An essentially equivalent development was made at the same time in England by Sir Geoffrey Taylor specifically for the propagation of earth waves away from an explosion. Subsequently, Taylor investigated the effect of removing the applied force. At the same time, and ignorant of the work of von Kármán and Taylor, LeVan Griffis and the writer investigated the effect of a constant impact force or impact velocity of finite duration applied to a very long uniform wire or bar.[5] In addition to giving the relation between impact velocity and the resulting stress and strain, and defining the critical impact velocity, this analysis showed that the final elongation must be considerably greater than that occurring at the end of impact, and that the resulting strain distribution is not a perfectly smooth function but contains a series of steps. These steps are caused by the alternating reflections or reverberations of the rapidly moving unstressing part (the back) of the stress-wave between the end of the specimen and the slowly moving plastic front. These discontinuities in residual strain distribution were predicted in this manner before being observed, or at least before they were recognized from experiments. Important contributions to the theory were made at this time by H. F. Bohnenblust and by C. Zener and J. H. Hollomon.

The propagation of plasticity in specimens of finite length was next studied, also independently, by von Kármán, Bohnenblust, and Hyers[6] at the California Institute of Technology and by Griffis and the writer[7] at the Illinois Institute of Technology.

The propagation of longitudinal waves in a medium having a stress-strain relation that is concave upward but with always positive slope was discussed by Griffis and the writer. This analysis was not intended to be applied to materials showing a decrease of stress with increasing strain (with upper and lower yield points) since it was felt that these might show a kind of unstable behavior; see, for example, Fig. 17 of the paper. The application was mainly for compression impact since in compression the resulting increase in cross-section combines with strain hardening to make the apparent stress increase faster than the strain sometimes, giving a concave upward stress-strain relation. Further investigations of compression impact were made by Griffis and the writer,[8] and resulted in determination of the conditions under which a specimen behaves essentially like a fluid, shooting off laterally under impact rather than remaining in contact with the impact hammer. A critical velocity of compression impact was defined as that at which this kind of behavior begins and was shown to be several times that for tension impact on the same material.

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[4] L. H. Donnell, "Longitudinal Wave Transmission and Impact," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 52, p. 153 (1930).

[5] M. P. White and L. Griffis, "The Permanent Strain in a Uniform Bar Due to Longitudinal Impact," NDRC Report A-71 (OSRD No. 742).

[6] Th. von Kármán, H. F. Bohnenblust, and D. H. Hyers, "The Propagation of Plastic Waves in Tension Specimens of Finite Length; Theory and Methods of Integration," NDRC Report A-103 (OSRD No. 946).

[7] L. Griffis, "The Behavior of Longitudinal Stress-waves near Discontinuities in Bars of Plastic Material," NDRC Report A-212 (OSRD No. 1799).

The problem of after-flow in short tension-impact specimens, that is, the amount of additional lengthening occurring after rupture due to the inertia of the material, was examined by the writer and, independently, by E. H. Lee, at that time of the Armament Research Department, Fort Halstead, England. It was found that the additional deformation produced in this way is not always negligible and may amount to several per cent. This also explained the double-necking sometimes observed in tension-impact specimens.

The principal theoretical problem of longitudinal impact yet to be solved concerns the behavior of a material with unstable deformation characteristics, that is, with an upper and a lower yield point. This is a particularly difficult problem since the rate of deformation is probably important and may have to be considered in the analysis.[9]

Before ending this discussion the writer wishes to make the following specific comments on the paper by Duwez and Clark:

In discussing Eq. 1 the authors state that it is necessary to assume that unloading follows a straight line of slope equal to Young's modulus for the material. Actually, the unloading characteristics of the material need not be considered at all unless the impact force is of finite duration, a case that the authors had not considered up to that point. Furthermore, when the effect of unloading is considered the particular slope chosen for the linear unloading relation does not affect the analysis. Unless the amount of deformation is large it is logical and in agreement with experiment to assume the unloading relation to be parallel to the elastic part of the loading relation, that is, of slope equal to the Young's modulus, but this assumption is not necessary.[10]

In discussing Fig. 7 the authors state that the velocity of propagation falls abruptly to zero at a strain of about 16 per cent. This is certainly shown on the figure but is not likely to be actually the case since that would require a discontinuity in the slope of the stress-strain curve near the maximum stress.

In discussing impact on wires of annealed low-carbon steel (Fig. 17) the authors state that a stress higher than the

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[8] M. P. White, "The Force Produced by Impact of a Cylindrical Body," NDRC Report A-153.

[9] Since writing this discussion the author has investigated the impact behavior of materials with definite yield points. The results are described in a paper to be published shortly.

[10] When the strain is large, the *apparent* Young's modulus during unloading—the ratio of increment of apparent stress σ to increment of engineering strain ϵ—is expected to differ from the Young's modulus during loading because of the change in dimensions of the specimen. Note that the analysis described in the paper under discussion is in terms of apparent stress, apparent Young's modulus, and engineering strain. If the *true* Young's modulus E', defined as the ratio of increment of actual stress to actual strain during an elastic deformation is assumed to be unaffected by any previous plastic deformation, then the apparent Young's modulus E will be given by the following expression:

$$E = E'/(1 + \epsilon)^2$$

During loading E and E' will be equal and during unloading they will differ according to the amount of plastic deformation produced. Since wave speed is proportional to $(E)^{\frac{1}{2}}$ the proportional change in wave speed will be equal and opposite to the amount of strain.

static yield point can be sustained for at least 2 or 3 milliseconds (without causing plastic deformation). Although the reason for selecting this figure is not given it is apparently equal to the duration of impact in the 42 ft. per sec. test. However, in this case there is a reflection from the fixed end of the specimen which travels with the elastic wave speed (since there is no plastic deformation anywhere ahead of it) and which therefore returns to the impact end of the specimen approximately 0.8 milliseconds after impact. The nature of this reflected stress-wave depends on what happens at the fixed end. If the stress at the fixed end is increased by reflection then the reflected wave will obviously be a wave of increased stress. This would be the case in a normal plastic or elastic material. But since the particular material under consideration can deform in an unstable manner, and since the impact stress exceeds the static yield stress, it seems to the writer that the strain that occurs at the fixed end when the wave is reflected there will actually result in a decrease of stress at that point, to a value below the incident stress. Should this be the case, the reflected wave becomes a wave of partial unstressing, which will reduce the stress at the impact end below its initial value. This happens, of course, about 0.8 milliseconds after impact. In support of the hypothesis that the fixed end reflection gives a reduction of stress in this case is the fact that the 42 ft. per sec. impact causes permanent strain only at the fixed and not at the impact end of the specimen.

A similar conclusion regarding the increase of yield strength under stresses of short duration was reached by Bertram Hopkinson in about 1905 from tests performed with falling weights and soft iron wires.[11]

In conclusion, the writer wishes to congratulate the authors on their presentation of a difficult and complicated subject. He hopes that this report will receive the attention that it deserves.

MESSRS. P. E. DUWEZ AND D. S. CLARK (*authors' closure by letter*).—The discussion by Mr. White for the most part is aimed at the origin of the theory of the propagation of plastic strain and not so much at the paper itself. This paper is a report of experimental results and is not concerned with the origin of the theory that they verify. As far as the authors are concerned, von Kármán proposed some experiments in July 1941 before a specific theory had been conceived. In discussing the first experimental results with von Kármán in September 1941, the authors were given his theory. On this basis, the experimental results presented in this paper were specifically directed to the von Kármán theory.

Mr. White has correctly directed attention to the needlessness of assuming that unloading follows a straight line of slope equal to Young's modulus of the material. Naturally, as long as no unloading takes place, any assumption about unloading is immaterial. The authors recognize that if unloading occurs, the theory can be carried through under more general assumptions.

The authors recognize that the derivative of the stress-strain curve is continuous and does not lead to a discontinuity in the velocity of propagation *versus* strain curve. However, in such curves, it is observed that the velocity of propagation decreases quite rapidly to zero and appears to fall abruptly. In drawing curves, such as Fig. 7, the abrupt fall was drawn as a straight line.

It is of interest to have Mr. Donnell refer to his previous short discussion of plastic wave transmission and to be able to give him experimental verification of his concept.

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[11] B. Hopkinson, "The Effects of Momentary Stresses in Materials," *Proceedings*, Royal Society London, Vol. 74, p. 498 (1905).

The propagation of plastic strain in an annealed low-carbon steel seems to be the most important question raised in the discussion. As pointed out in the paper, the theory of propagation of plastic waves with a concave upward curvature and in particular to a material for which a portion of the stress-strain curve is horizontal (yield portion). Mr. Donnell seems to think that in such case, the superposition method has the advantage of providing a solution. His discussion is vague. The authors believe that his intuitive reasoning is the same as that stated more explicitly by Mr. Griffis, who discusses the problem further by assuming that when there is a yield point, a so-called shock wave propagated with a finite velocity. The authors wish to insist on the fact that the existence of a shock wave in annealed mild steel is hypothetical. If this hypothesis is accepted, Mr. Griffis' analysis is perfectly correct. Unfortunately, the experimental results clearly indicate that the assumption of a shock wave cannot explain the actual behavior of the metal. A careful reading of the paper should make this point apparent, but the following additional evidence is given in order to clarify this point.

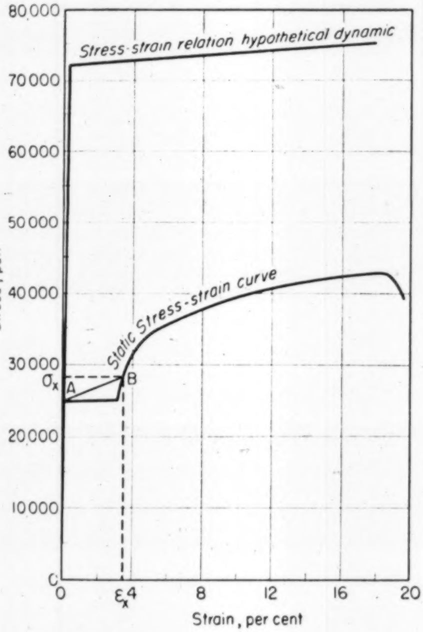

FIG. 23.—Static Stress-Strain Curve and Hypothetical Dynamic Stress-Strain Relation for Annealed Low Carbon Steel (Wire).

A stress-strain curve of the annealed low-carbon steel wire used in the experiments is presented in Fig. 23. Assuming the process of propagation involves a shock wave when the impact velocity is above 14 ft. per sec. (corresponding to the value $v = \epsilon_0 c_0$) a plastic wave would propagate with a velocity corresponding to the straight line AB. The value of a plastic strain ϵ_x is in such case given by $v_0 = \epsilon_0 c_0 + \epsilon_x c_x$ (1) in which c_x is the velocity of propagation of the plastic shock. This velocity is given by $\sqrt{\frac{\sigma_x - \sigma_0}{\epsilon_x} \frac{1}{\rho}}$. Equation 1, and the condition that the point B must be on the stress-strain curve, determines the value of c_x. Solving the problem by approximation, it is found that for a velocity of 42 ft. per sec. (which is the maximum impact velocity at which no plastic strain occurs at the moving end), the value of ϵ_x would be 3.3 per cent and the velocity of propagation of the shock would be 825 ft. per sec. For impact velocities between 14 ft. per sec. and 42 ft. per sec., a plastic strain between 3.1 and 3.4 per cent should propagate from the moving end (3.1 per cent being the yielding strain). These strains are not small, as inferred by Mr. Griffis, and would have been detected, since the accuracy of the measurements performed on the iron wire with the comparator was within 0.002 in. per inch (0.2 per cent) as stated on page 508.

By assuming a shock wave theory, the

plastic strain should propagate from the moving end as soon as the impact velocity exceeds the value corresponding to the static yield point. A large number of tests in tension as well as in compression were performed on annealed low-carbon steel, and in no case did a plastic strain occur at the moving end for impact velocities less than from 2 to 3 times the velocity corresponding to the yield point. Furthermore, as explained on page 521, the velocity of propagation of the plastic strain was found to be practically independent of the strain. It seems, therefore, logical to the authors to explain the behavior of the material on the basis of a raised yield point and a practically straight and slightly inclined line in the range of plastic deformation as indicated in curve 2 of Fig. 23. In such case, there is no need for a theory based on the assumption of a plastic shock wave.

Another controversy concerns the statement that "a stress higher than the static yield point can be sustained for at least 2 or 3 milliseconds after the passage of the wave." Even under the assumption of a raised yield point, there are not five elastic reflections, since plastic deformation is observed, and the stress at the moving end never reaches, as Mr. Griffis states, the value of 216,000 psi. (by his analysis should be 360,000 psi.). If the original strain at the moving end is less than $\epsilon_0'/2$ (ϵ_0' being the strain corresponding to the raised yield point) the stress at the moving end remains elastic until the elastic portion of the loading wave reflected from the fixed end comes back to the moving end and then plastic strain should occur. The time during which an elastic stress greater than the static yield stress can be sustained by the specimen near the moving end is therefore at least twice the time it takes for the elastic wave to travel the length of the specimen, namely, approximately 0.8 milliseconds as computed by Mr. White. Experimentally however, no plastic strain was found near the moving end in tests made with impact velocities between 14 and 42 ft. per sec. and yet the duration of impact was between 2 and 3 milliseconds. The authors recognize that this behavior cannot be explained if the assumption of a dynamic stress-strain curve as shown by curve 2 in Fig. 23 is made. Mr. White offers the explanation of instability phenomena, which might occur at the fixed end. Mr. White's explanation may be fully justified, but a more exact formulation of what effect this instability has on the wave propagation would be required in order to proceed further with the analysis. The authors believe that different experimental data should be secured with iron specimens so that additional light may be cast upon the very puzzling phenomenon of dynamic yielding.

The authors are very appreciative of the extensive discussion of this paper and are pleased to note the interest shown in this subject.

THE VELOCITY ASPECT OF TENSION-IMPACT TESTING*[1]

By William H. Hoppmann II[2]

Synopsis

This paper reviews briefly the work done by various investigators of the velocity aspect of tension-impact testing and, in particular, describes the Guillotine Impact Testing Machine provided by the Navy at the Material Laboratory, N. Y. Naval Shipyard, Brooklyn, N. Y., for the study of the effect of moderately high velocities on the impact resistance of materials and minor structural elements.

An exposition is also given of the method of Theodore von Kármán (**9**)[3] for dealing with the propagation of plastic deformations in solids, together with his concept of "critical velocity."

Utilizing the von Kármán method, the critical velocity for a hard-drawn copper has been calculated. The analysis is given along with experimental verification by test results from the Guillotine Impact Machine.

When it is considered that it has taken nearly half a century to progress as far as we have in impact testing with oversimplified assumptions and very hazy knowledge of how such variables as temperature, shape, size, and velocity determine impact resistance of materials and of structures it should be very clear that much effort and ingenuity are yet required to provide worth-while knowledge for designers and engineers faced with the problem of impact.

Within the last ten years or so, starting with the very suggestive work of H. C. Mann at Watertown Arsenal, the theory of impact, particularly from the standpoint of velocity, has been greatly advanced and the knowledge gathered in that period may reasonably be said to constitute the sum total of our knowledge of the subject today.

The greatest advance in theory and test technique has come through the efforts of the physicists and engineers working in the field of applied mechanics (**2**, **4**, **12**). However, this important field of technical activity is not and should not be a small closed corporation with regard to the development of impact testing. It is well known that the standard impact test for the purpose of determining the effects of heat treatment was introduced by the metallurgist at the beginning of the century. Since those days the scope and content of the subject have expanded vastly. Metallurgists themselves are being spurred to re-examine the foundations of their work involving impact testing. The fine and capable investigations reported by Kahn and Imbembo (**19**) in their paper describing the effects of shape and temperature in the Charpy impact test show what can be done by the metallurgist in re-evaluating the meaning of the impact test. Such work is a contribution toward establishing a comprehensive foundation of knowledge for the proper study of impact which ultimately redounds to the benefit of the designer.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Presented at the Bureau of Ships Metallurgical Conference, held at the Material Laboratory, N. Y. Naval Shipyard, Brooklyn, N. Y., September 25, 1945. The opinions or assertions contained herein are the private ones of the author and are not to be construed as official or reflecting the views of the Navy Department or the Naval Service at large.

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[3] The boldface numbers in parentheses refer to the references appended to this paper, see p. 543.

HIGH VELOCITY IMPACT TESTING MACHINES AND GUILLOTINE IMPACT MACHINE AT BROOKLYN, N. Y.

About fifteen years ago H. C. Mann developed a rotating disk type of

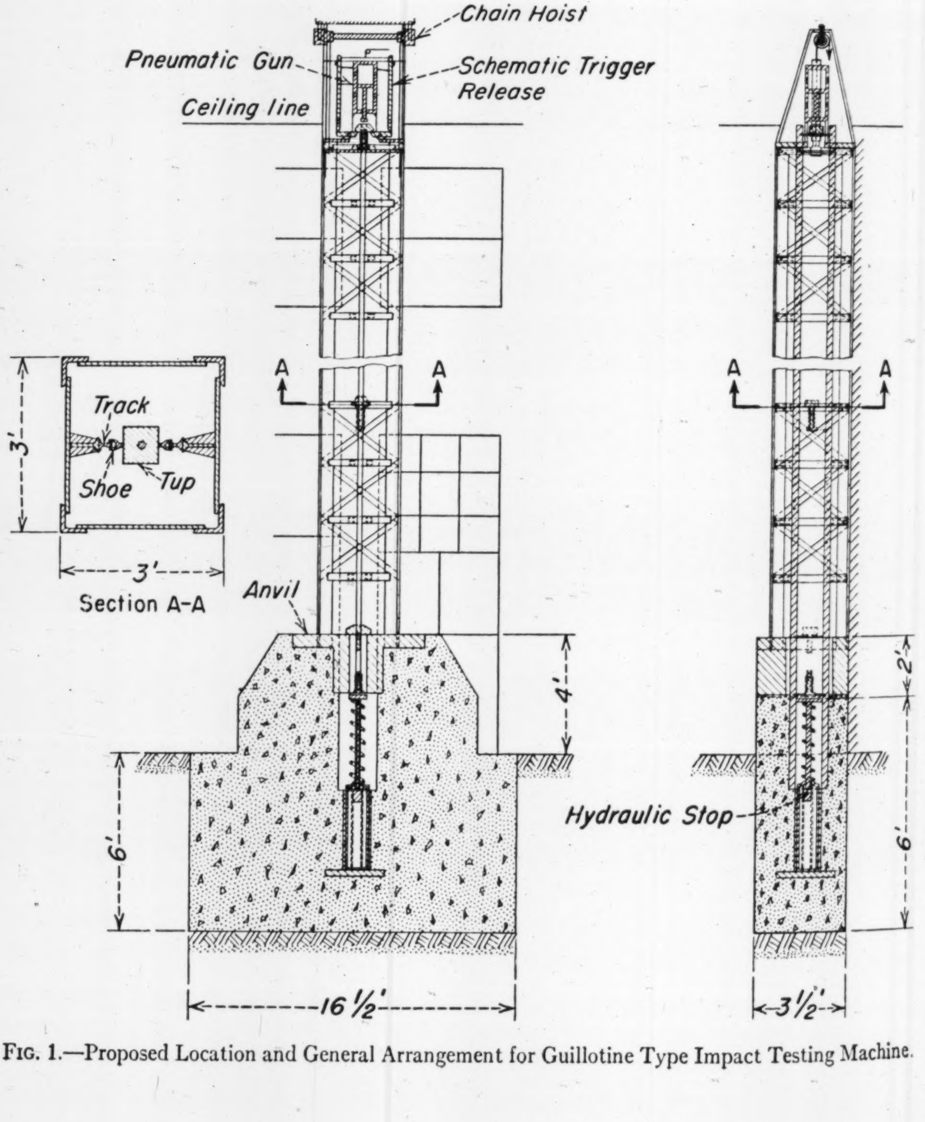

Chain Hoist
Pneumatic Gun
Schematic Trigger Release
Ceiling line
A
A
Track
Shoe
Tup
3'
3'
Section A-A
Anvil
4'
6'
16 1/2'
A
A
2'
Hydraulic Stop
6'
3 1/2'

FIG. 1.—Proposed Location and General Arrangement for Guillotine Type Impact Testing Machine.

variable speed impact testing machine (1). Linear impact speeds up to several hundred feet per second were obtained. Since that time a commercial company has manufactured a similar machine which has been used by various laboratories. Machines of the same general type have been built and used by Nadai (4) at the Westinghouse Laboratories and by experimenters at the California Institute of Technology (2).

During the past five years a Guillotine Impact Testing Machine was designed and manufactured by the N. Y. Naval Shipyard, Brooklyn, N. Y. (6). It was then installed in the Material Laboratory and has been under development to produce the highest speed and most intense impacts consistent with the design and strength of the machine.

The machine is approximately 80 ft. high. It consists essentially of a tower with a pneumatic gun at the top and an anvil at the bottom. The tower is provided with steel rails for guiding a test assembly consisting of a tup or hammer below which a tension specimen hangs with a weight attached to the bottom of the specimen. A spacing collar is placed, concentric with the specimen, between the hammer and the weight. The assembly either is dropped freely under the force of gravity or is fired from the pneumatic cylinder at the top of the tower. A split anvil near the bottom of the tower intercepts the hammer but permits the weight to fall through an opening. In this manner the specimen is loaded by the weight pulling on one end and the hammer bouncing off the anvil at the other end. After rupture of the specimen the weight comes to rest on an hydraulic buffer at the base of the machine and the hammer comes to rest on the anvil. A schematic diagram of the machine is shown in Fig. 1.

As originally planned, the machine was to develop a maximum impact speed of 100 ft. per second. Since additional knowledge has become available that "critical" velocities for many metals of interest to the Navy are in the range between 100 and 200 ft. per second at temperatures near 70 F., effort has been made to increase the maximum velocity obtainable with the Guillotine Machine. So far specimens have been tested up to 125 ft. per second. Because of the fact that the machine is designed to test large specimens of high impact resistance, the appurtenances necessarily have to have high weight-to-strength ratios. The present hammer weighs about 30 lb., and when this strikes the anvil with a velocity in excess of 100 ft. per second it approaches its ultimate strength and failure will ensue. A different type hammer with a considerably lower weight-to-strength ratio has been designed and it may give velocities between 150 and 200 ft. per second.

The machine has been used to test specimens that require 10,000 ft-lb. (15) to cause failure, and it is considered that 15,000 ft-lb. may be available if required. Tests have been conducted in which specimens have absorbed less than 100 ft-lb. (17). This is a very large range of the test variables, and considerable caution is required to produce satisfactory accuracy.

In order to obtain force-time data for a specimen there is provided in series with the test specimens, between the hammer and the weight, a weigh bar to which resistance wire strain gages are attached (14). When the specimen is being loaded to rupture, after the hammer strikes the anvil, the gages measure the strain in the weigh bar at each instant of time and accordingly give the force pulling on the specimen. For this purpose, electric contacts in the form of insulated copper strips are attached to the tower near the guide rails for the entire height. The electric impulses are taken off the copper contact strips at the top of the tower and fed through amplifiers and control devices to a cathode-ray oscilloscope where they are recorded. Sliding copper shoes, attached to the falling assembly, slide on the copper contact strips in order to form a continuous circuit at all times.

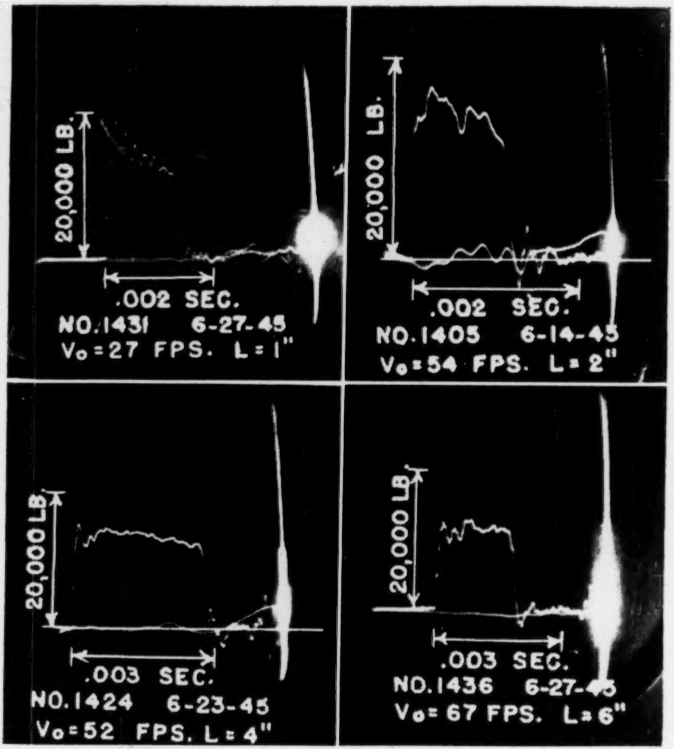


FIG. 2.—Force-Time Oscillograms of Medium Steel (0.505-in. Diameter) on the Guillotine Impact Machine.

Typical force-time oscillograms obtained on the Guillotine machines are shown in Fig. 2.

For the purpose of determining the energy absorbed in a specimen by measuring the velocity of the hammer and of the weight, a synchronous spark recorder has been developed and put into use (**15**). The sparks are made to discharge between needles on the hammer and weight and high voltage plates, on which are fastened wax-coated papers. The wax melts when the sparks flow and small holes are then burnt in the paper. The sparks are generated at a rate of 180 per second. As the spark needles are located on the parts of the specimen assembly of which the velocities are to be measured, the series of melted spots on the recording wax papers provides displacement-time records from which velocities can readily be deduced. A schematic arrangement illustrating the spark-recording device is shown in Fig. 3.

VON KÁRMÁN THEORY OF IMPACT: VERIFICATION ON THE GUILLOTINE IMPACT TESTING MACHINE

Within the last few years, Theodore von Kármán under the auspices of the National Defense Research Council conducted an investigation of the theory of the tension-impact test (9). In connection with the work, he described a method of dealing with plastic waves in bars under tension and thereby directly provided the rational basis for the study of impact involving plastic deformation and the reason for the velocity effect in the tension-impact test noted by investigators.

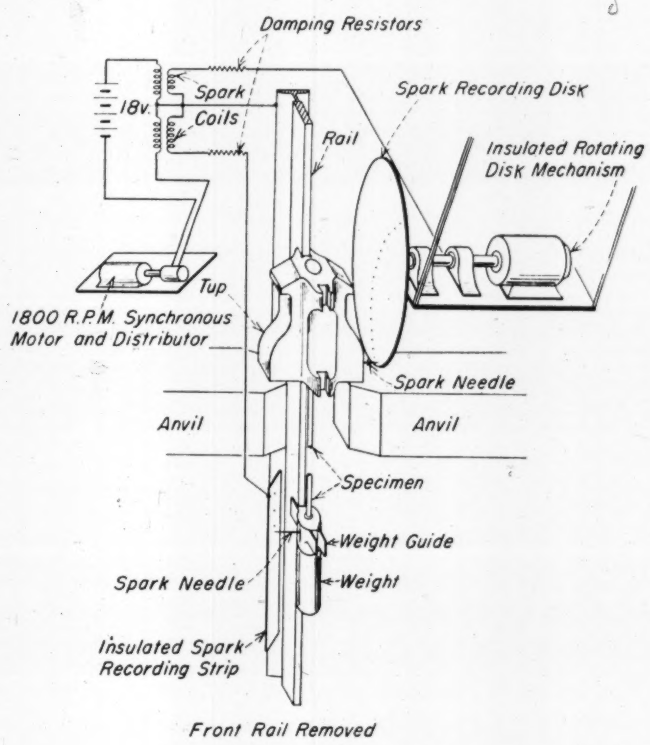

Damping Resistors
18v.
Spark Coils
Rail
Spark Recording Disk
Insulated Rotating Disk Mechanism
Tup
1800 R.P.M. Synchronous Motor and Distributor
Spark Needle
Anvil
Anvil
Specimen
Weight Guide
Weight
Spark Needle
Insulated Spark Recording Strip
Front Rail Removed

FIG. 3.—Schematic Arrangement of Timed Spark Velocity Measuring Device, Guillotine Impact Testing Machine.

One of the most noteworthy results of his work was the demonstration of a reason for the notion of "critical velocity" of impact from the standpoint of classical mechanics. In practice, the critical velocity of impact is that for which energy of absorption and elongation decrease sharply.

As has been known for many years and exhibited in text books on the subject—"Theory of Elasticity" by Timoshenko, for example—the phenomena of stress and strain in a bar can be studied for the purely elastic case in the form of a differential equation subject to given boundary and initial value conditions.

The equation is:

$$\frac{\partial^2 u}{\partial t^2} = c^2 \frac{\partial^2 u}{\partial x^2}$$

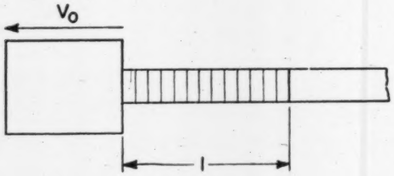
where:

u = displacement at point in a bar,
x = co-ordinate of that point,
c = velocity of sound in bar, and
t = time.

It is well known that the constant c is $\sqrt{\frac{E}{\rho}}$ where E is Young's modulus of elasticity and ρ is the density of the material.

A simple use of this theory enables one to compute directly the stress in a bar having its end subjected to a velocity V_o *provided the elastic limit of the material is not exceeded.*

Consider a bar with one end moving with a velocity V_o. In any time, t, a stress wave will move into the bar a definite distance and by the condition of continuity (no break in the material) the end will move a distance equal to the total elongation of the strained portion of the bar as follows:



now

$$V_o t = \epsilon \cdot l = \epsilon \cdot c \cdot t$$

or

$$V_o = \epsilon \cdot c$$

and

$$\epsilon = \frac{V_o}{c}$$

further

$$\sigma = \epsilon \cdot E = E \cdot \frac{V_o}{c}$$

where:

V_o = velocity of end of the rod,

l = distance stress wave travels in time t,

t = time,

σ = stress,

ϵ = strain (uniform throughout distance l),

E = Young's modulus, and

c = velocity of stress wave (acoustical velocity).

As an example, for steel
$E = 30 \times 10^6$ and
$c = 17{,}000$ ft. per second.
For a velocity of impact of 20 ft. per second, the stress immediately after impact will be

$$\sigma = \frac{20}{17{,}000} \times 30 \times 10^6 = 35{,}300 \text{ psi.}$$

When the yield point of the material is exceeded, this simple method is no longer applicable. Extensions of the ideas back of this type of analysis, while leading to more complicated analytical expressions, yield the rational basis for the concept of "critical velocity" as applied to impact.

Von Kármán's main contribution consists in the extension of the ideas to the case of plasticity. Instead of the constant slope E of the standard engineering stress-strain curve, the variable slope $\frac{\partial\sigma}{\partial\epsilon}$ in the plastic region where σ is the stress and ϵ the strain, is used. The differential equation corresponding to the case of plastic deformations is then as follows:

$$\frac{\partial\sigma}{\partial\epsilon} \cdot \frac{\partial^2 u}{\partial x^2} = \rho \frac{\partial^2 u}{\partial t^2}$$

This equation can be solved subject to the boundary and initial value conditions by use of the engineering stress-strain curve.

In order to illustrate the method of analysis for determining the "critical velocity" based on the plastic wave concept, the data obtained in impact tests on a hard-drawn copper tested in the Guillotine Impact Machine of the N. Y. Naval Shipyard, Brooklyn, N. Y., will now be used (16).

So that the phenomenon of reflection may be dispensed with and the analysis simplified, consider a semi-infinite bar with its end subjected to a constant velocity V_o. In accordance with the differential equation of motion, a strain wave front will move along the bar. The portion of the wave that has moved farthest into the bar at any time will be elastic strain and it travels with a velocity equal to $\sqrt{\frac{E}{\rho}}$ where E is Young's modulus of elasticity and ρ is the mass density. It is followed by plastic strains moving with less velocity. The velocity of a plastic wave is equal to $\sqrt{\frac{\partial\sigma}{\partial\epsilon}\Big/\rho}$ where $\frac{\partial\sigma}{\partial\epsilon}$ is the slope of the engineering stress-strain curve and ρ is the mass density.

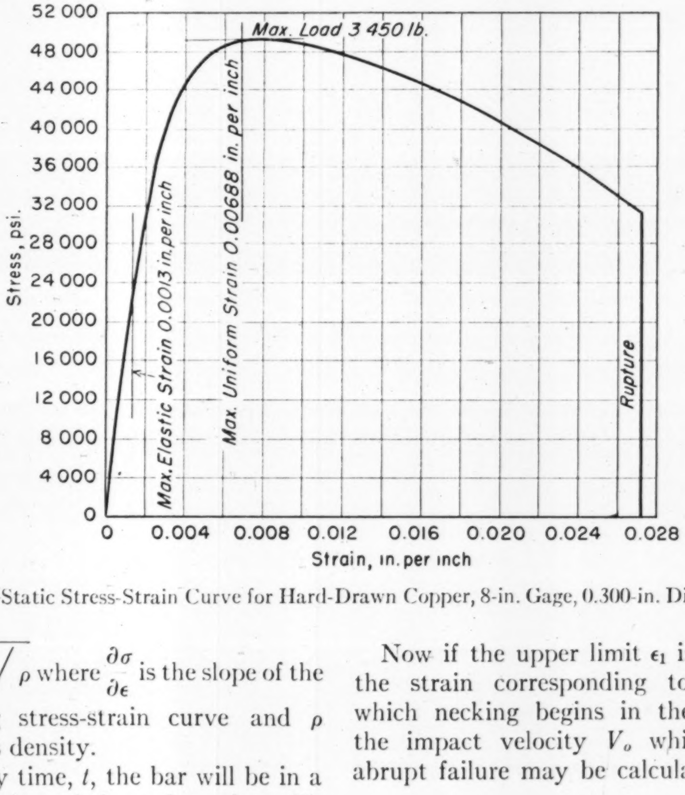


FIG. 4.—Static Stress-Strain Curve for Hard-Drawn Copper, 8-in. Gage, 0.300-in. Diameter.

After any time, t, the bar will be in a state of strain and the end moving with a velocity V_o will have moved through a displacement $V_o t$. This displacement must be equal to the total elongation of the bar at the time t. A strain of magnitude ϵ will move to a point in the bar $V(\epsilon)t$, or X, distant from the end at which the impact is applied. The total elongation will then be

$$e = \int_0^{\epsilon} V(\epsilon)t d\epsilon = \int_0^{x} \epsilon_x dx$$

And so

$$V_o t = t \int_0^{\epsilon_1} V(\epsilon)\, d\epsilon$$

or

$$V_o = \int_0^{\epsilon_1} V(\epsilon)\, d\epsilon$$

Now if the upper limit ϵ_1 is taken as the strain corresponding to that at which necking begins in the bar, ϵ_m, the impact velocity V_o which causes abrupt failure may be calculated from:

$$V_o = \int_0^{\epsilon_m} V(\epsilon)\, d\epsilon$$

The value of the integral can be calculated from the static stress-strain curve for the material. Figure 4 gives this curve for the case of hard-drawn copper. Since $V(\epsilon) = \sqrt{\frac{\partial\sigma}{\partial\epsilon}\Big/\rho}$ a curve of $V(\epsilon)$ as a function of ϵ may be constructed with the aid of the stress-strain curve by obtaining the slopes $\frac{\partial\sigma}{\partial\epsilon}$ for several values of strain ϵ. This curve for the copper is shown in Fig. 5. Now the area under the curve is equal to the value of $\int_0^{\epsilon_m} V(\epsilon)d\epsilon$, and hence the "critical velocity" is given as 47.3 ft. per second. The areas for various values of ϵ are plotted in Fig. 6.

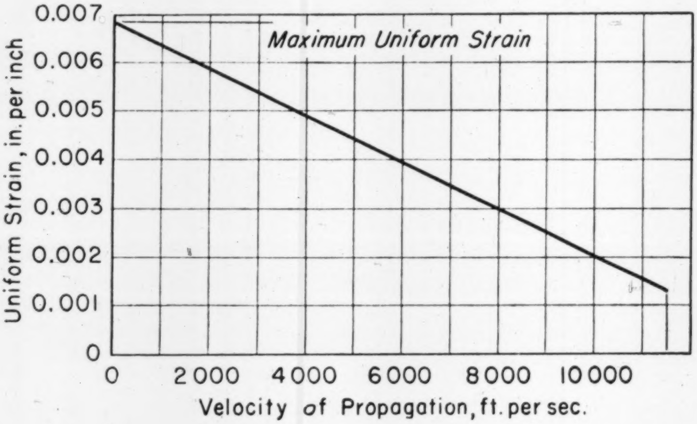


FIG. 5.—Variation of Strain with Velocity of Propagation for Hard Copper, 8-in. Gage, 0.300-in. Diameter.

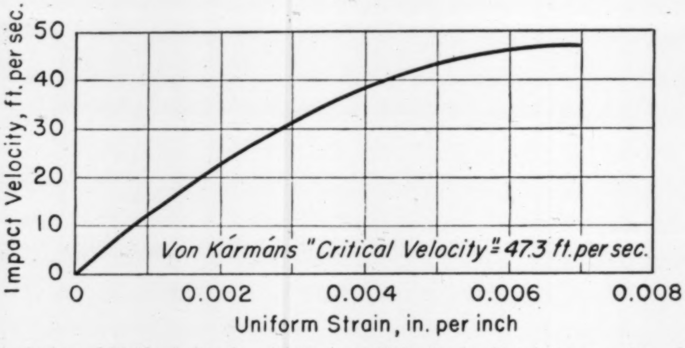


FIG. 6.—Variation of Uniform Strain with Impact Velocity for Hard Copper, 8-in. Gage, 0.300-in. Diameter.

The variations of energy and total elongation with velocity of impact are shown in Figs. 7 and 8, respectively. It may be noted that a sharp decrease in energy and in elongation occurs at about 55 ft. per second which approximately checks the theoretical value of "critical velocity" calculated by the von Kármán method.

In the above analysis no mention was made of the particle velocity in the bar. This velocity is known to be, in general, less than the wave velocity. It may be instructive to note that at the moving end of the bar it is equal to the velocity of impact. Furthermore, there is a definite relation between the particle velocity at any point in the bar and the train of strain waves that have passed the point. Assume that at time zero the front of the strain wave just reaches a point, P, in the bar. Then after a time, t, the wave of disturbance will have passed beyond the point and a state of

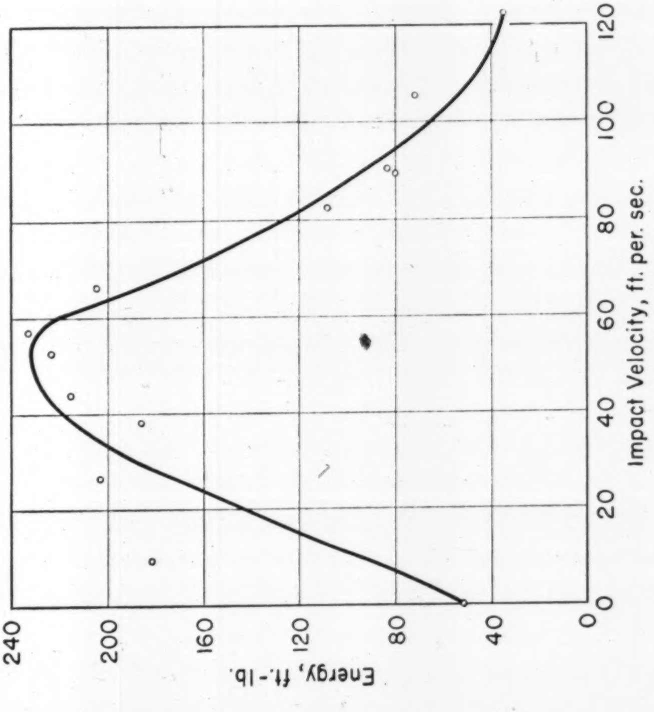


FIG. 7.—Variation of Energy with Impact Velocity for Hard Copper, 8-in. Gage, 0.300-in. Diameter.

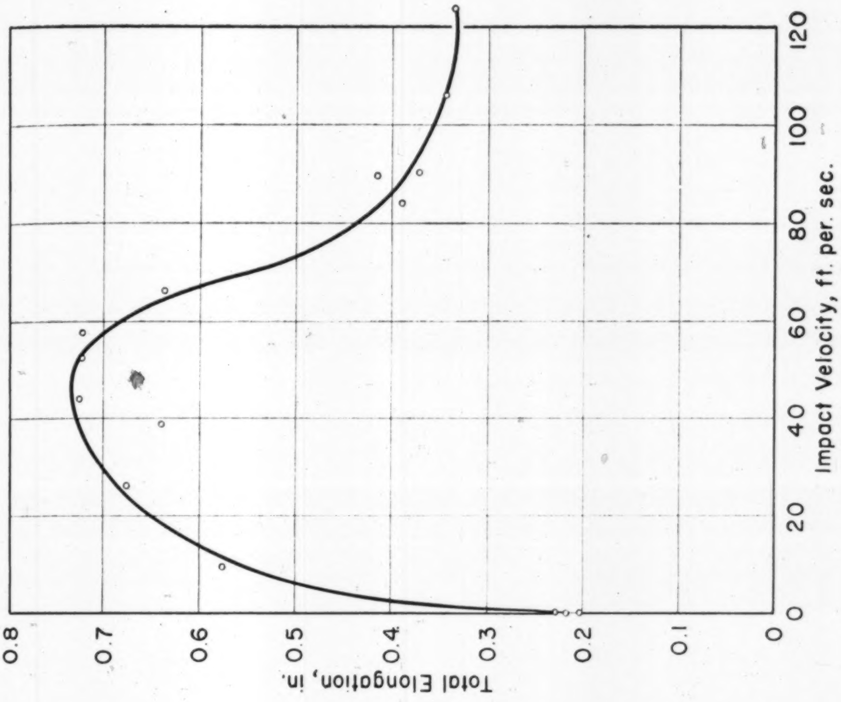


FIG. 8.—Variation of Elongation with Impact Velocity for Hard Copper, 8-in. Gage, 0.300-in. Diameter.

strain will have been built up from zero to ϵ_1. To each strain ϵ there corresponds a wave velocity $V(\epsilon)$.

The particle velocity of the point is then

$$V = \int_0^{\epsilon_1} V(\epsilon)\, d\epsilon$$

Note the identity of this expression with that which gives the "critical velocity."

An important consequence of this result is that velocity $V_o = \int_0^{\epsilon_m} V(\epsilon) d\epsilon$ is critical and precipitates failure in the material regardless of the manner in which the velocity on the end of the bar is applied. It should be recalled that in the von Kármán solution V_o was assumed constant. Now it can be seen that it may vary and that if and when V_o is reached the bar fails with low energy of absorption and small elongation. Again, ϵ_m is the strain at which necking begins in the ordinary tension specimen.

It should be noted that on the basis of the von Kármán method presented above the nature of the stress-strain curve alone determines the value of the "critical velocity." In general, if the curve should change it may be expected that the "critical velocity" will change. In particular, if the stress-strain relation for the material depends on temperature the "critical velocity" will also depend on temperature and a functional relation will subsist amongst the physical variables—temperature, velocity of impact, and energy of absorption. It is known that the stress-strain relation varies with temperature and consequently so does the "critical velocity." A few impact data exist in the literature to substantiate this point. More extensive work to determine accurately the relationship is planned for the near future.

CONCLUSION

High-speed impact testing machines have been developed which demonstrate that a "critical velocity" exists which if exceeded in tension-impact tests will cause failure characterized by low energy of absorption and small total elongations (**16**, **10**). In addition it is clear that von Kármán has indicated the broad basis in classical mechanics which explains the nature of the effect of high velocity on materials in the tension-impact test (**9**, **10**, **11**, **12**).

It is likewise obvious that while a beginning has been made toward the rational explanation of phenomena attendant on failure in impact, there remain many questions to be answered as well as analytical and experimental tools to be developed in order to extend our knowledge of the many phases of impact to test and design.

For example, it is known that a change in temperature of test causes a shift in the "critical velocity" (**3**). A complete study of the effect of velocity on failure due to impact loads must involve a study of temperature effect. By many investigators of the impact test it has been shown that for a given velocity of test, energy of absorption and "brittleness" vary with the temperature (**3**, **4**). Now it is known that energy of absorption, elongation, and "brittle" nature of fracture vary as functions of temperature, velocity, shape, and size. (**1**, **4**, **7**, **8**, **10**).

Von Kármán in connection with his analytical work carefully points out that the stress-strain curve in the dynamic condition may be different from that in the static condition. Also in actual test specimens reflection of the strain waves from the ends of the specimen must be taken into account (**11**).

Notwithstanding these observations, however, it can be seen that data obtained on the Guillotine machine with hard-drawn copper agreed well with the result predicted on the basis of the von Kármán theory for the semi-infinite bar. That means that in principle we are on the right track and that further work will be toward extension of principles and clarification of details.

While the discussion in this paper has been devoted to the tension-impact test, it is easy to see that the same fundamental basis for velocity effects in that type of test taken from the field of classical mechanics also exists for analysis of the Charpy and of the Izod tests.

It is hoped that the Navy will not only continue its interest and effort in studies of impact both in application to test and design but will expand and intensify them. The way and spirit are illustrated in a thoughtful report written by Captain W. P. Roop, U. S. Navy, Retired, for the David W. Taylor Model Basin in 1943 (**18**).

Finally it is concluded that the metallurgist, the engineer, and the physicist are making definite contributions to the knowledge of impact which are of basic importance to the designer of structures. That knowledge intimately concerns the mechanism of propagation of deformation by plastic waves in the materials and structures as well as the extent of deformation. In addition, there is being compiled a vast amount of usable information about the important variables of velocity and temperature accompanying impact as well as of the effects of size, shape, and structure. Ultimately, the rational application of this knowledge, when integrated, will enable the designer really to utilize the enormous impact resistance of materials. This has not been done up to the present.

Acknowledgments:

I wish to thank my associates Messrs. E. C. Taylor and F. Lager of the Material Laboratory, N. Y. Naval Shipyard, Brooklyn, N. Y., for their important assistance in developing the Guillotine Impact Testing Machine.

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DISCUSSION

Mr. Howard Scott.[1]—I should like to ask Mr. Hoppmann whether or not the peak in the energy or the elongation curve plotted against impact velocity in a function of the fracture type, that is, whether or not there is a change in the fracture appearance as the velocity is increased above that producing maximum elongation.

Mr. William H. Hoppmann (*author's closure by letter*).—In reply to the question of Mr. Scott, I should like to say that there was no noticeable change in appearance of fracture as a function of velocity. However, the importance of his question is clear to me and I think that micrographic studies should be made of the structure of the metal in order to determine effects of the velocity of impact.

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PLASTIC FLOW OF A MAGNESIUM ALLOY UNDER BIAXIAL STRESSES*[1]

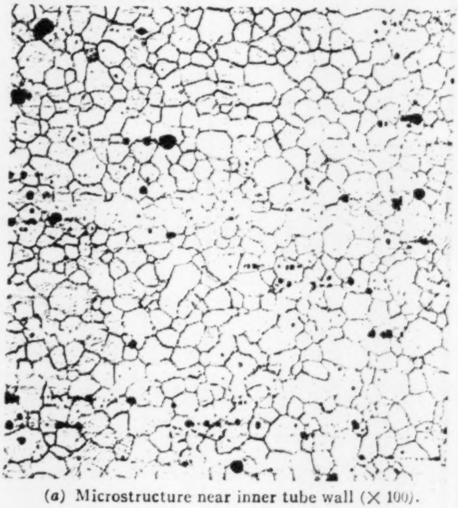
By D. M. Cunningham,[2] E. G. Thomsen,[2] and J. E. Dorn[2]

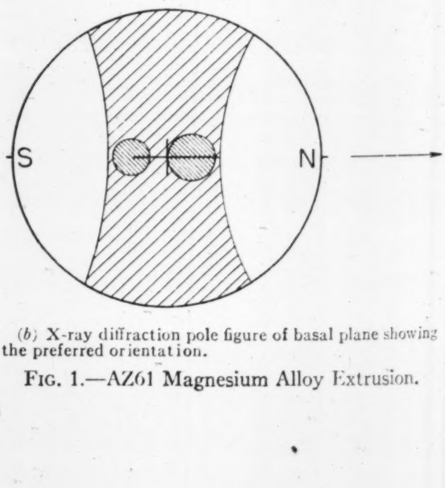
Numerous investigations (1-9)[3] have been made of the effect of combined stresses on the plastic flow of metals. An extensive series of references pertaining to this subject can be found in published monographs and reports (10-16).

Early experimental studies of the plastic flow of metals under combined stresses were generally conducted under conditions of biaxial stressing for constant ratios and directions of the principal stresses. Analytical procedures for describing the plastic flow of metals when the stress-ratios vary (6, 7, 13) and general methods of analysis appropriate for examples where the directions of principal stresses vary with deformation (13, 17-20) are now available. The present paper is principally directed to an experimental evaluation of the plastic flow of AZ61 magnesium alloy extrusion under varying ratios of biaxial stressing. The quota data were presented in Office of Scientific Research and Development Report No. 4172 (21).

Materials, Equipment, and Method

A tubular extrusion of AZ61 alloy, which has a typical composition of 6.5 per cent Al, 0.2 per cent Mn, and 0.7 per cent Zn, was used in this study. The microstructure shown in Fig. 1(*a*), reveals fairly uniform equiaxed fine grains. The pole figure, Fig. 1(*b*), however, indicates a fairly high degree of preferred orientation suggesting that the metal is initially anisotropic. Nevertheless, the reported data reveal that the isotropic


(*a*) Microstructure near inner tube wall (× 100).



(*b*) X-ray diffraction pole figure of basal plane showing the preferred orientation.

Fig. 1.—AZ61 Magnesium Alloy Extrusion.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] From Thesis submitted by D. M. Cunningham in partial fulfillment of the requirements for the M. S. Degree.

[2] Lecturer, Assistant Professor, and Associate Professor of Engineering, University of California, Berkeley, Calif.

[3] The boldface numbers in parentheses refer to the references appended to this paper, see p. 552.

theory of plastic flow in polycrystalline metals may be applied with fair accuracy to this material. Some deviations between the experimental facts and the theory, however, are attributable to initial anisotropy.

The design of the test specimen and the method of stressing were fully described in an earlier report (**5**) and need not be completely repeated here. The specimen, gages, and loading fixtures are shown in Fig. 2. The inside diameter of the tubular specimen was 0.375 in. and the gage section was carefully machined to a diameter of 0.437 in. The conventional procedure of applying an axial load and internal pressure was used in order to obtain combined axial and circumferential stresses. Strains were measured by axial and diametral gages, from which the longitudinal and circumferential strains were calculated. The radial strains were calculated from the measured axial and circumferential strains by means of the equation for constancy of volume.

THEORY

A formal theory of plastic flow in metals (**7, 13, 17, 18**) has been based on the following assumptions:

1. The material is homogeneous and continuously isotropic.
2. The increments of strain are linear functions of the stresses.
3. The change in volume during finite plastic deformation is negligible.
4. The flow stress is independent of the strain path and time and depends only on the plastic strain energy.

Although none of these assumptions is in complete agreement with the physical facts, they lead directly to an idealized theory which has the undeniable merit of simplicity and utility when compared to more exact theories which attempt to account for anisotropy, nonlinearity of the stress-strain relationships, change in volume, and other second order phenomena such as time effects and the Bauschinger effect.

This idealized theory requires that (see explanation of symbols in Appendix I)

$$\left.\begin{aligned}
{}^P\epsilon_{zz} &= \frac{d\,{}^P\bar{\phi}}{\bar{\sigma}}\,[\tau_{zz} - \tfrac{1}{2}(\tau_{rr} + \tau_{\theta\theta})], \\
{}^P\epsilon_{rr} &= \frac{d\,{}^P\bar{\phi}}{\bar{\sigma}}\,[\tau_{rr} - \tfrac{1}{2}(\tau_{\theta\theta} + \tau_{zz})], \\
{}^P\epsilon_{\theta\theta} &= \frac{d\,{}^P\bar{\phi}}{\bar{\sigma}}\,[\tau_{\theta\theta} - \tfrac{1}{2}(\tau_{zz} + \tau_{rr})], \\
{}^P\epsilon_{zr} &= 3\,\frac{d\,{}^P\bar{\phi}}{\bar{\sigma}}\,\tau_{zr}, \\
{}^P\epsilon_{r\theta} &= 3\,\frac{d\,{}^P\bar{\phi}}{\bar{\sigma}}\,\tau_{r\theta}, \qquad {}^P\epsilon_{\theta z} = 3\,\frac{d\,{}^P\bar{\phi}}{\bar{\sigma}}\,\tau_{\theta z},
\end{aligned}\right\} \ldots (1)$$

where, $\bar{\sigma}$, the effective stress for plastic flow is

$$\bar{\sigma} = \sqrt{\frac{(\tau_{zz} - \tau_{rr})^2 + (\tau_{rr} - \tau_{\theta\theta})^2 + (\tau_{\theta\theta} - \tau_{zz})^2}{2} + 3(\tau_{zr}^2 + \tau_{r\theta}^2 + \tau_{\theta z}^2} \,\ldots\ldots\ldots (2)$$

and ${}^P\bar{\phi}$, the effective plastic strain, is

$${}^P\bar{\phi} = \frac{2}{3}\int \sqrt{\frac{({}^P\epsilon_{zz} - {}^P\epsilon_{,r})^2 + ({}^P\epsilon_{rr} - {}^P\epsilon_{\theta\theta})^2 + ({}^P\epsilon_{\theta\theta} - {}^P\epsilon_{zz})^2}{2} + \tfrac{3}{4}({}^P\epsilon_{zr\theta}^2 + {}^P\epsilon_r^2 + {}^P\epsilon_{\theta z}^2)}. \ldots\ldots (3)$$

The work-hardening curve is given by

$$\bar{\sigma} = \bar{\sigma}(\bar{\phi}) = \bar{\sigma}\left[{}^P\bar{\phi} + \frac{2(1 + \nu)}{3E}\,\bar{\sigma}\right] \ldots (4)$$

where $\bar{\phi}$ is the total effective strain, which is the sum of the plastic effective strain and the contribution of the elastic effective strain, namely,

$$\bar{\phi} = {}^P\bar{\phi} + {}^E\bar{\phi} = {}^P\bar{\phi} + \frac{2(1 + \nu)}{3E}\,\bar{\sigma}. \ldots (5)$$

EXPERIMENTAL EVALUATION OF STRESSES AND STRAINS

When a thin-walled tubular specimen is loaded by internal pressure and an axial load, the principal stresses coincide with the axial, circumferential, and radial directions. Consequently the shear stresses, τ_{zr}, $\tau_{r\theta}$, $\tau_{\ z}$ vanish, and, by Eq. 1, the shear strains also vanish.

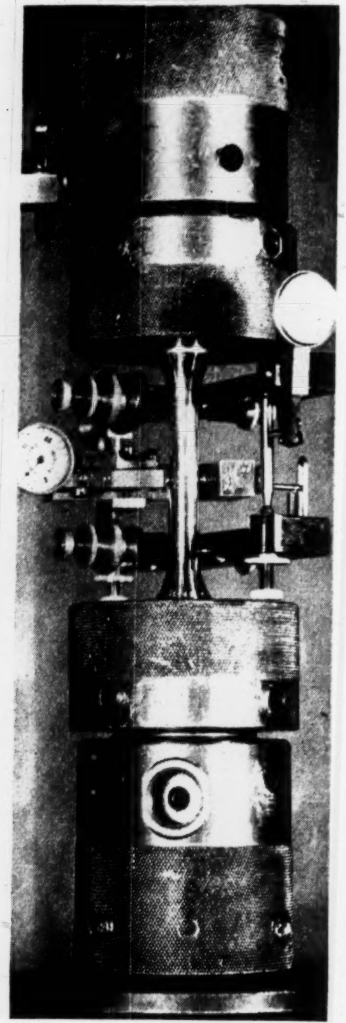

FIG. 2.—Tubular Specimen in Grips Before Loading Showing Position of the Extensometers.

Since the principal directions of stress and strain coincide at all times with the axial, circumferential, and radial directions, fibers initially aligned in these directions continue to remain in these directions. Thus the instantaneous infinitesimal changes of finite strain on such fibers are

$$d\,{}^P\phi_{zz} = {}^P\epsilon_{zz} = d \ln (l/l_o) \ldots\ldots\ldots\ldots (6)$$

$$d\,{}^P\phi_{rr} = {}^P\epsilon_{rr} = d \ln \left(\frac{R_o - R_i}{R_{oo} - R_{io}}\right) \ldots (7)$$

$$d\,{}^P\phi_{\theta\theta} = {}^P\epsilon_{\theta\theta} = d \ln (R_o/R_{oo}) \ldots\ldots\ldots (8)$$

and the finite strains on these fibers at any instant of the deformation are obtainable by direct integration of these equations yielding

$${}^P\phi_{zz} = \ln (l/l_o) \ldots\ldots\ldots\ldots\ldots\ldots (9)$$

$${}^P\phi_{rr} = \ln \left(\frac{R_o - R_i}{R_{oo} - R_{io}}\right) \ldots\ldots\ldots (10)$$

$${}^P\phi_{\theta\theta} = \ln (R_o/R_{oo}) \ldots\ldots\ldots\ldots (11)$$

where all quantities are experimentally determinable except R_i. The value of R_i can be obtained by adding the first three of the six Eq. 1, yielding the condition of constancy of volume, ${}^P\epsilon_{zz} + {}^P\epsilon_r + {}^P\epsilon_{\theta\theta} = 0$, or

$${}^P\epsilon_{rr} = d \ln \left(\frac{R_o - R_i}{R_{oo} - R_{io}}\right)$$

$$= -({}^P\epsilon_{\theta\theta} + {}^P\epsilon_{zz}) = d \ln \left(\frac{l_o R_{oo}}{l R_o}\right) \ldots (12)$$

whence, upon integration,

$${}^P\phi_{rr} = -({}^P\phi_{zz} + {}^P\phi_{\theta\theta}) \ldots\ldots\ldots (13)$$

and,

$$R_i = R_o - (R_{o0} - R_{io}) \left(\frac{l_o R_{oo}}{l R_o}\right) \ldots (14)$$

The value of ${}^P\phi$ is then obtainable by integration of Eq. 3, following the evaluation of the strains as given in Eqs. 6, 7, 8, and 12. The normal stresses in the thin-walled tube can be calculated directly from the well-known formulas:

$$\tau_{zz} = \frac{L + P\pi R_i^2}{\pi(R_o^2 - R_i^2)} \quad \ldots\ldots (15)$$

$$\tau_{\theta\theta} = \frac{PR_i}{(R_o - R_i)} \quad \ldots\ldots (16)$$

$$(\tau_{rr})_{\text{ave.}} = -P/2 \quad \ldots\ldots (17)$$

wherein all quantities are determinable.

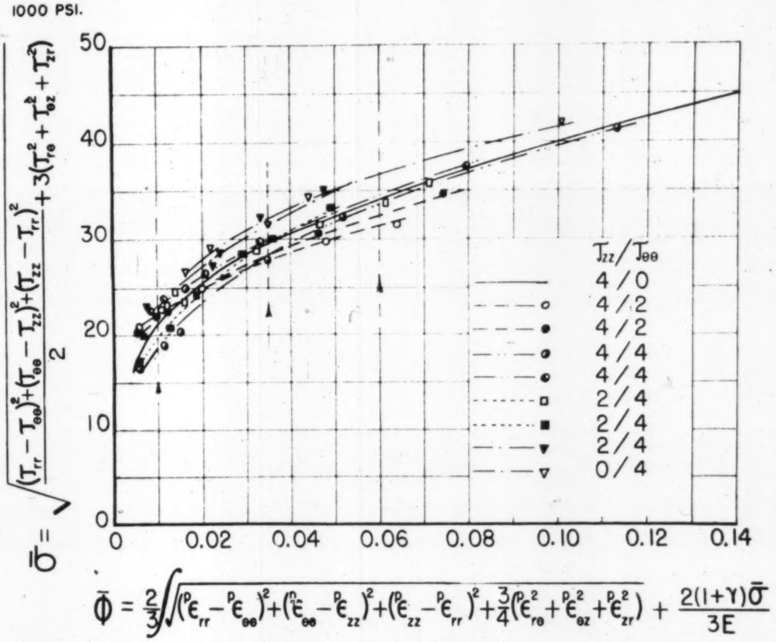

FIG. 3.—Effective Stress-Effective Strain Curves for AZ61 Magnesium Alloy Extrusions in the Tension-Tension Field of Biaxial Stresses.

The value of the effective stress, $\bar{\sigma}$, at any instant of the deformation of the tube can now be obtained by introducing the experimentally determined values of the stresses given by Eqs. 15, 16, and 17 into Eq. 2.

CONSTANT STRESS RATIOS

The conventional investigations of plastic flow under substantially biaxial stresses are generally conducted so that the ratios of stress

$$\tau_{\theta\theta}/\tau_{zz} = \alpha,$$

$$\text{and } \tau_{rr}/\tau_{zz} = \beta = (\text{a small quantity}) \ldots (18)$$

are maintained fairly constant throughout the deformation.[4] Therefore,

$$\bar{\sigma} = \tau_{zz}\sqrt{\frac{(1-\alpha)^2+(\alpha-\beta)^2+(\beta-1)^2}{2}} \quad \ldots (19)$$

Under conditions of constant stress ratios, the strain ratios also remain constant as revealed by applying Eq. 1; and

$$\left.\begin{aligned} {}^P\epsilon_{\theta\theta}/{}^P\epsilon_{zz} &= \left\{\frac{\alpha - \frac{1}{2}(\beta+1)}{1-\frac{1}{2}(\alpha+\beta)}\right\} \\ &= {}^P\phi_{\theta\theta}/{}^P\phi_{zz} = A \\ \frac{{}^P\epsilon_{rr}}{{}^P\epsilon_{\theta\theta}} &= \left\{\frac{\beta - \frac{1}{2}(1+\alpha)}{1-\frac{1}{2}(\alpha+\beta)}\right\} = \frac{{}^P\phi_{rr}}{{}^P\phi_{zz}} = B \end{aligned}\right\} (20)$$

Introducing Eq. 20 into Eq. 3 and integrating, gives

$${}^P\bar{\phi} = {}^P\phi_{zz}$$

$$\frac{2}{3}\sqrt{\frac{(1-A)^2+(A-B)^2+(B-1)^2}{2}} \quad \ldots (21)$$

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[4] Note.—if τ_{zz} is zero, another principal stress must be used for the denominator.

or, by reapplying Eq. 20,

$$^{P}\bar{\phi} = \frac{2}{3}\sqrt{\frac{(^{P}\phi_{zz} - {}^{P}\phi_{\theta\theta})^2 + (^{P}\phi_{\theta\theta} - {}^{P}\phi_{rr})^2 + (^{P}\phi_{rr} - {}^{P}\phi_{zz})^2}{2}} \quad \ldots\ldots\ldots\ldots(22)$$

The experimental results obtained for various ratios of $\tau_{zz}/\tau_{\theta\theta}$ on tubular extrusions of AZ61 alloy are given in Fig. 3. A metal which obeys the idealized theory of plastic flow should have provided a single curve, whereas the actual data deviate about ± 7 per cent from a mean curve. This variation is attributable to differences between the assumptions in the theory and the physical facts, and suggests the need for a more comprehensive theory.

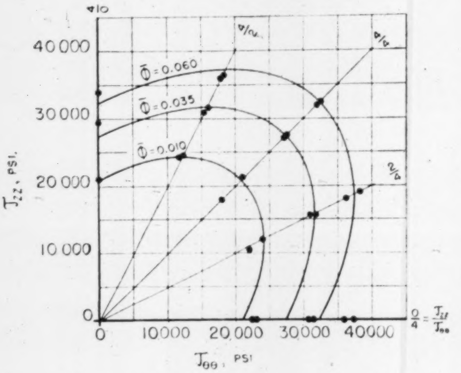

FIG. 4.—Biaxial Stress Ellipse for Constant Effective Stress and Effective Strain.

The values of τ_{zz} and $\tau_{\theta\theta}$ for various values of $^{P}\phi$ are shown in Fig. 4. At a given value of $^{P}\bar{\phi}$, $\bar{\sigma}$ should be constant. If τ_{rr} is negligible, a factor generally valid for thin walled tubes,

$$\bar{\sigma}^2 = \frac{(\tau_{zz} - \tau_{\theta\theta})^2 + \tau_{\theta\theta}^2 + \tau_{zz}^2}{2} \quad \ldots.(23)$$

Thus τ_{zz} and $\tau_{\theta\theta}$ trace an ellipse, with its major axis at 45 deg. to the τ_{zz} and $\tau_{\theta\theta}$ axes in the biaxial stress plot. The solid curves represent the theoretical ellipses for several values of $^{P}\phi$.

Again the agreement of the theory with the facts is imperfect and the experimental values lie between those determined by the plastic strain energy and the maximum shear stress theories. Nevertheless, the data contained in Fig. 4 illustrate the utility of the simple theory for first order estimation of facts.

VARIABLE STRESS RATIOS

It will prove interesting now to consider a more general case of biaxial stressing for which the stress ratios do not remain constant. One method of obtaining this condition consists of stressing first in simple tension to a value $\tau_{zz} = \tau_A$, $(\tau_{\theta\theta} = \tau_{rr} = 0)$ and subsequently increasing $\tau_{\theta\theta}$ to τ_B by increasing the pressure, maintaining $\tau_{zz} = \tau_A$ constant by proper adjustment of the load. The specific stress path used in this investigation is shown in Fig. 5. For purposes of illustration, however, it will be assumed that a $\bar{\sigma} = \bar{\sigma}\ (^{P}\phi)$ curve has been obtained by a previously performed tension test or some other simple method and the evaluation of the finite strains at the conclusion of the test is desired.

The finite strains at the conclusion of the test are obtained by the integration of Eqs. 1 over the entire path of deformation according to the following proposed plan:

Path	Stresses
	$\tau_{rr} = \tau_{\theta\theta} = 0$;
$0 - A$	τ_{zz} varies from 0 to τ_A
$A - B$	$\tau_{rr} = 0$; $\tau_{zz} = \tau_A$;
	$\tau_{\theta\theta}$ varies from 0 to τ_B

Thus, over path A to B, the stress ratio $\tau_{\theta\theta}/\tau_{zz}$ varies from zero to τ_B/τ_A. Integration of Eq. 1 for the present example, yields

$$^{P}\phi_{\theta\theta} = \int_0^{^{P}\bar{\phi}_A} (-\tfrac{1}{2}\tau_{zz})\frac{d\,^{P}\bar{\phi}}{\bar{\sigma}} + \int_{^{P}\bar{\psi}_A}^{^{P}\bar{\phi}_B} (\tau_{\theta\theta} - \tfrac{1}{2}\tau_A)\frac{d\,^{P}\bar{\phi}}{\bar{\sigma}} \quad \ldots.(24)$$

and

$$^{P}\phi_{zz} = \int_0^{^{P}\bar{\phi}_A} \tau_{zz} \frac{d\,^{P}\bar{\phi}}{\bar{\sigma}} + \int_{^{P}\bar{\phi}_A}^{^{P}\bar{\phi}_B} (\tau_A - \tfrac{1}{2}\tau_{\theta\theta}) \frac{d\,^{P}\bar{\phi}}{\bar{\sigma}} \quad (25)$$

Over the path from 0 to A, $\bar{\sigma} = \tau_{zz}$, and over the path from A to B

$$\bar{\sigma} = \sqrt{\tau_{\theta\theta}^2 - \tau_A \tau_{\theta\theta} + \tau_A^2} \quad (26)$$

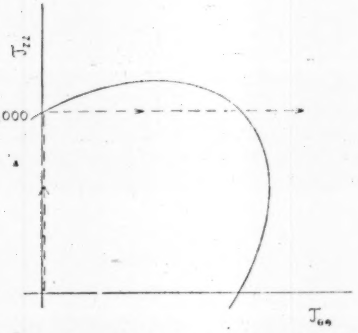

FIG. 5.—Stress Path Where $\tau_{\theta\theta} = 0$, while τ_{zz} Is Increased to 28,000 psi. Then with τ_{zz} maintained at 28,000 psi., $\tau_{\theta\theta}$ is increased until rupture occurs.

Therefore

$$^{P}\phi_{\theta\theta} = -\tfrac{1}{2}\,^{P}\bar{\phi}_A + \int_{^{P}\bar{\phi}_A}^{^{P}\bar{\phi}_B} \left\{ \frac{\sqrt{\bar{\sigma}^2 - \frac{3}{4}\tau_A^2}}{\bar{\sigma}} \right\} d\,^{P}\bar{\phi} \quad (27)$$

$$^{P}\phi_{zz} = \,^{P}\bar{\phi}_A + \int_{^{P}\bar{\phi}_A}^{^{P}\bar{\phi}_B} \cdot \left\{ \frac{\frac{3}{4}\tau_A - \sqrt{\frac{1}{4}(\bar{\sigma}^2 - \frac{3}{4}\tau_A^2)}}{\bar{\sigma}} \right\} d\,^{P}\bar{\phi} \quad (28)$$

The integrations over any range $^{P}\phi_A$ to $^{P}\phi_B$ may be obtained by plotting the integrand of the above integrals from $^{P}\phi_A$ to the desired $^{P}\phi_B$ as a function of $^{P}\phi$ and integrating graphically. The values of this integrand are determinable from the effective stress-effective strain curve which may be obtained from a simple tension test and the value of τ_A.

The points in Fig. 6 are experimentally determined finite strains for tubular magnesium alloy extrusion when stressed in simple tension to 28,000 psi. and then stressed in the circumferential direction holding $\tau_{zz} = \tau_A$ at 28,000 psi. The graphical integration described in the previous paragraph was made from the solid $\bar{\sigma} - {}^{P}\phi$ curve of Fig. 3 which had been previously obtained by simple tension stressing. The predicted strains are shown by the solid line of Fig. 6.

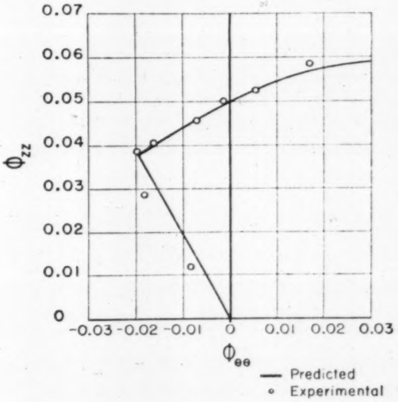

FIG. 6.—Strain Path for the Stress Path Where τ_{zz} Is Increased from 0 to 28,000 psi. and Then Held Constant While $\tau_{\theta\theta}$ Is Increased from 0 to the Rupture Stress.

During the first stage of tensile stressing, the circumferential strain deviated slightly from the theoretical value $^{P}\phi_{\theta\theta} = -\frac{1}{2}\,^{P}\phi_{zz}$ required for truly isotropic metals. The remainder of the curve, however, exhibits remarkably good agreement between theoretical predictions and the facts.

CONCLUSIONS

1. The idealized theory of plastic flow of work-hardenable metals provides a useful method of describing plastic phenomena in metals under conditions where the work-hardened state is only a function of plastic work and is insensitive to temporal effects.

2. The agreement between the idealized theory and the facts is imperfect. The deviations arise from numerous causes such as inhomogeneities, anisotropy, nonlinear stress-strain relations, anelastic effects such as the Bauschinger effect and elastic aftereffects, and temporal effects resulting from strain aging and recrystallization. Additional investigations are required to isolate these factors and determine their function in a more general theory of plastic flow of metals.

3. In the range of its intended application, the agreement of the idealized theory of metal plasticity with the experimental facts reveals correct trends and relative orders of magnitude to provide a useful approximate method of analysis.

4. Current methods of analysis have been extended to cases where the principal directions of stress rotate and where the stress ratios may vary. In the present paper an example of a case of varying stress ratio was analyzed.

APPENDIX I

SYMBOLS

τ_{zz} = the normal stress in the axial direction of a tube.

$\tau_{\theta\theta}$ = the normal stress in the circumferential direction.

τ_{rr} = the normal stress in the radial direction.

$\tau_{r\theta}$, $\tau_{\theta z}$, τ_{zr} = shear stresses.

${}^{P}\phi_{zz}$, ${}^{P}\phi_{\theta\theta}$, ${}^{P}\phi_{rr}$ = finite plastic strains corresponding to normal stresses.

${}^{P}\epsilon_{zz}$, ${}^{P}\epsilon_{\theta\theta}$, ${}^{P}\epsilon_{rr}$ = infinitesimal plastic strains.

${}^{P}\epsilon_{r\theta}$, ${}^{P}\epsilon_{\theta z}$, ${}^{P}\epsilon_{zr}$ = infinitesimal plastic shear strains.

ν = Poisson's ratio.

E = Young's modulus.

L = axial load on the tube.

P = internal pressure in the tube.

R_{i0} = inside radius of tube before deformation.

R_{o0} = outside radius of tube before deformation.

R_i = inside radius of the tube at any stage of deformation.

R_o = outside radius of the tube at any stage of deformation.

l_0 = length of tube before deformation.

l = length of tube after deformation.

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INFLUENCE OF PLASTIC EXTENSION AND COMPRESSION ON THE FRACTURE STRESS OF METALS[*]

By D. J. McAdam, Jr.,[1] G. W. Geil,[1] and W. D. Jenkins[1]

Synopsis

According to a prevalent view, plastic deformation has practically no effect on the fracture stress of any metals except pearlitic steels and metals with similar microstructure. Moreover, the prevalent view is that the effect of plastic deformation on the fracture stress of pearlitic steels is anisotropic. This paper presents results of longitudinal and transverse tests on cold-extended steel plate and of tension tests on previously compressed cylindrical steel bars. The results show that the general effect of plastic deformation on the fracture stress is a work-hardening effect similar to the effect on the flow stress. They also lead to the conclusion that any kind of plastic deformation of any polycrystalline metal affects the fracture stress in the same way that it affects the corresponding flow stress.

The term "fracture stress" is here used in the same sense as "technical cohesion limit." It designates the technically determinable resistance to fracture. In a series of papers, the authors and their associates have shown that the technical cohesion limit is affected by the same four factors that affect the flow stress,[2] namely, the stress system, plastic deformation, temperature, and the strain rate (**9** to **20**).[3] Moreover, the influence of each of these factors on the technical cohesion limit has been shown to be qualitatively similar to the influence of the same factor on the flow stress. The influence of any one of these factors may be represented by a curve of cohesion limits, although only one point on the curve may represent actual fracture. As the term "fracture stress" has come into use with the same significance as "technical cohesion limit," the authors are here using the shorter term, with the understanding that it does not always refer to actual fracture.

According to the prevalent views, the conditions determining the fracture stress differ greatly from those affecting the flow stress, and are much more complex (**1** to **6**, **21** to **24**). In the opinion of the authors, these views are based on misinterpretation of the evidence, principally because of preconceived views about how metals "should" behave. One of these ideas is that the stress at "brittle" fracture is a material constant, unaffected by the stress system and by plastic deformation, and even unaffected by temperature and the strain rate. This idea probably is the origin of the prevalent view that fractures can be classified as either brittle or ductile, and that the two classes are subject to different laws.

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[2] Flow stress is here used in the generally accepted significance to designate the greatest principal stress during flow.

[3] The boldface numbers in parentheses refer to the references appended to this paper, see p. 566.

This view has been elaborated by Orowan (21). After classifying fractures as either brittle or ductile, he subdivides ductile fractures into fibrous fractures and shear fractures, and says that they are subject to different laws. Moreover, he proposes a fourth type of fracture, in which the metal draws down to a point. Since the fourth type probably should be viewed as extreme plastic deformation rather than as fracture, and since the stress at the sharp point cannot be determined, this type can be dismissed from consideration in a study of the factors affecting the fracture stress.

It is not generally made clear whether the alleged distinction between brittle and ductile fractures is based on the plastic deformation before fracture or on the deformation during fracture. On either basis, the authors have not found a boundary between brittle fracture and fibrous fracture. A rough distinction can be made between crystalline fractures and transverse dull fractures. Sometimes, however, the surfaces of fracture are speckled with crystalline and dull spots. Moreover, the plastic deformation before fracture sometimes is much less for a dull fracture than for a crystalline fracture, and the deformation during fracture sometimes is no less for a crystalline fracture than for a dull fracture. In the investigation of thousands of fractures with ductility ranging from very high values to values too small to be detected by ordinary means, the authors have found the influence of any of these factors on the fracture stress to be qualitatively the same, whether the metal is brittle or ductile.

In tension tests of notched or unnotched specimens, fracture generally starts as a transverse crack, which extends outward from the axis. The crack may extend thus entirely to the periphery, or it may change to an oblique shear fracture, thus forming the familiar cup and cone. The change from transverse to oblique fracture probably never occurs except in a notched specimen or after the beginning of the local contraction of an unnotched specimen. The change probably never occurs under unidirectional tension, but may occur under a combination of longitudinal and transverse tensions. Orowan considers that the change from transverse to shear fracture is due to the fact that the available energy is insufficient for further extension of the transverse crack (21). He supposes that the plastic deformation during shear fracture somehow facilitates the progress of the fracture. In the author's opinion, however, the change from transverse to oblique fracture is due to a change in the stress system during the spreading of the transverse crack. When the metal is soft or is inherently very ductile, the extension of the transverse crack eventually causes a decrease in the radial stress ratio (ratio of transverse to longitudinal stress) and in the stress concentration at the edge of the crack. This change in the stress system causes an abrupt increase in the ductility of the metal ahead of the crack. As fracture proceeds during the rapid increase in ductility, it might be expected to take an oblique course. When the metal is harder and less ductile, the change in the stress system is not such as to increase the ductility, and the crack extends transversely to the periphery. Even when the crack starts during local contraction, the fracture sometimes changes from dull to crystalline, thus indicating that the ductility of the metal has decreased as the crack extended.

Whether or not the fracture changes from transverse to oblique, and whether the ductility is small or great, the fracture stress is definitely determinable, and the study of the factors affecting the fracture stress need not be confused by distinctions between types of fracture.

For many years there has been evidence in the literature that plastic deformation increases the fracture stress. This evidence was discussed in the first two of the previously mentioned series of papers (9, 10). The evidence is based on results of tension tests of notched specimens. Comparison of fracture stresses of metals that had been cold-worked before formation of the notch, with fracture stresses of metals that had not been cold-worked, revealed the influence of two factors, namely, plastic deformation and the stress system. The evidence showed that the fracture stress increases with plastic extension and with the radial stress ratio. In succeeding papers by the authors and their associates (11 to 17, 19, 20) there is much additional evidence based on tension tests of notched specimens of many ferrous and non-ferrous metals, and it has been shown that plastic extension increases the fracture stress of all these metals. The fact that both plastic deformation and the stress system affect the fracture stress in the same way that they affect the flow stress, suggested that the effect of plastic deformation on the fracture stress is a work-hardening or strengthening effect, similar to the effect on the flow stress. This idea received support when it was found later that temperature (12 to 16, 20) and the strain rate (18) affect the fracture stress of many metals in the same way that they affect the flow stress.

Evidence of a different kind was obtained by McAdam and Mebs in 1943 (12). By comparing the breaking stresses of cold-worked and annealed ingot iron at −188 C., they found that the prior cold work had greatly increased the stress required for brittle fracture. The results were regarded as merely confirming the evidence already obtained with notched specimens.

Since publication of these results Hollomon and Zener (3) reported results of experiments in which pearlitic steels were cold-worked various amounts at room temperature and then tested to fracture at −188 C. The results indicated that the breaking stress at −188 C. increased continuously with the prior plastic deformation at room temperature. They reported, however, that steels with microstructure consisting of tempered martensite showed no evidence of increase in the fracture stress. To explain this difference in behavior, they assumed that the effect of plastic extension on the fracture stress of pearlitic steels is a purely anisotropic effect, due to reorientation of the internal flaws, which are generally believed to account for the great difference between the theoretical and actual breaking stresses of metals. The internal flaws in steels were assumed to be similar in form to the cementite particles. Reorientation of the lamellar flaws in pearlite was supposed to increase the fracture stress, whereas reorientation of the spheroidal flaws in tempered martensite was supposed to have no effect on the fracture stress. That this interpretation is not valid, however, is indicated by the previously published evidence that plastic extension increases the fracture stress of ingot iron, which is practically free from cementite particles (12).

The idea that any effect of plastic deformation on the fracture stress is purely anisotropic has been amplified and presented repeatedly by Hollomon (2 to 6). According to his view, plastic deformation has no effect on the fracture stress of single-phase metals (6). It is not made clear why reorientation of the internal flaws that are supposed to be in all metals would affect the fracture stress of a pearlitic steel but not the fracture stress of a single-phase metal. In support of the view that plastic extension of pearlitic steel lowers the fracture stress in transverse directions, Hollomon (2 to 6) repeatedly mentions results of experiments by Swift (23) in which bars of steel were twisted various amounts and then fractured by tension. When the twist exceeded a certain amount, both the fracture stress and the ductility decreased. However, the decrease in the fracture stress may be attributed principally to the decrease in the amount of plastic deformation at which fracture occurs, owing to a slight differential effect of the torsion on the curves of flow and fracture. The results of these experiments do not exclude the possibility that twisting the specimens increased the cohesive strength in all directions.

As additional support for the idea that plastic extension lowers the fracture stress in transverse directions, Hollomon refers to the unusual fractures obtained with some steels during the local contraction of tension test specimens (5, 6). The crack first extends along the axis of the specimen, then curves around to a transverse direction. The specimen thus may break into three or more pieces. Hollomon says that the only possible explanation of such a fracture is that the plastic extension of the specimen has lowered the fracture stress in transverse radial directions. The authors, however, have long regarded such fractures as illustrations of the fact that fracture is determined by the entire stress system, not by the stress in any one direction. A fracture of this type merely reveals a slight anisotropy due to the initial microstructure. Because of this anisotropy, the increase of the fracture stress with plastic extension was somewhat less in the transverse direction than in the longitudinal direction.

In support of the view that compression lowers the tensile fracture stress Hollomon (6) mentions results of experiments by Bridgman (1) and by Körber, Eichinger, and Möller (7, 8). His conclusion is that "as the deformation increases in compression, the tensile stress (in the opposite direction) required for fracture decreases. These results indicate that the effect of compressive deformation is exactly opposite to the effect of tensile deformation." Results of experiments to be presented in this paper, however, show that this conclusion is not correct.

The experiments described in this paper were designed to determine whether the effect of plastic deformation on the fracture stress is merely anisotropic or is a work-hardening effect similar to the effect on the flow stress. The first series of experiments consisted of tension tests of longitudinal and transverse specimens from hot-rolled steel plate and from plate that had been extended cold by tension. The second series consisted of tension tests of specimens cut from cylindrical bars that had been compressed various amounts. Tension tests were made at room temperature and in liquid air (−188 C.).

MATERIALS, SPECIMENS, AND METHODS OF TESTING

The steel plate for the investigation of the influence of plastic extension on fracture under transverse tension was supplied by the David Taylor Model Basin of the Navy Department, Washington, D. C., through the cooperation of D. F. Windenburg and H. R. Thomas. The chemical composition of the steel is given in Table I. Two pieces 6 ft. long, 9 in. wide, and 1 in. thick were extended by tension in the same direction as the extension by hot rolling. The following is from the report of the procedure by the Model Basin:

"In producing the plastic elongation, the load was applied to incipient necking and then released. Elongation during loading was measured over the full length of the specimens by use of a special extensometer. Scribed transverse lines placed 10 in. apart over the central 30 in. of the length permit determining the plastic deformation at different parts of the length. The elongations over the three 10-in. gage lengths agreed with each other to less than 0.05 per cent."

Only the central 30-in. length was used in the series of experiments to be described. The plastic extension was found to be 11.3 per cent, and the decrease in width was nearly equal to the decrease in thickness. Two pieces 1 ft. square and 1 in. thick were also supplied in the hot-rolled condition.

For the longitudinal and transverse tension tests of the steel plate, specimens with cylindrical cross-section and 2-in. gage length were prepared; the threaded ends were 0.75 in. in diameter. For the tests at room temperature, the gage diameter was 0.505 in.; for the tests in liquid air, the diameter was smaller, about 0.4 in., in order to avoid fracture in the threaded ends.

TABLE I.—CHEMICAL COMPOSITION.

Material	Designation	Chemical Composition, per cent					
		C	Mn	P	S	Si	Cr
Ship Plate.........	BD	0.26	0.50	0.018	0.025	0.03	...
0.45 per cent C, 0.90 per cent Cr Steel.	CS	0.45	0.62	0.022	0.012	0.24	0.90

Annealed chromium steel (Table I) was used for the investigation of the influence of compression followed by extension. Since the flow stress at the yield point of the annealed steel dropped only slightly, less than 100 psi., this steel seemed more suitable for compression tests than an annealed carbon steel. The steel was in the form of cylindrical bars $2\frac{1}{4}$ in. in diameter. By using specimens about 7 in. long for the compression tests, it was possible to obtain satisfactory specimens for the subsequent tension tests. Before the compression tests, this steel was normalized at 1650 F. (900 C.) and annealed for 2 hr. at 1550 F. (840 C.). The specimens were then machined so as to produce smooth cylindrical surfaces, with the ends perpendicular to the axis.

The compression tests were made by the Engineering Mechanics Section of the National Bureau of Standards with the assistance of L. R. Sweetman. The testing machine used was a horizontal Emery machine of 2,300,000-lb. capacity. The five specimens used were compressed different amounts; one of these was compressed to incipient buckling. During the tests frequent measurements were made of the changes in length and diameter. The changes in diameter were measured at midlength and at three other places along the specimen. After completion of a compression test, diameter measurements were made at nine places along the specimen and in four directions at each place. Table II gives the initial and final diameters at midlength and the equivalent decreases in length.

TABLE II.—COMPRESSION TESTS.

Original Diameter, in.	Final Diameter, in.	Decrease in Length, per cent	A_0/A*
2.191.............	2.231	3.6	0.964
2.188.............	2.265	6.8	0.932
2.205	2.319	9.6	0.904
2.198.............	2.341	11.9	0.881
2.197.............	2.399	16.2	0.838

* A_0 = Initial sectional area.
A = Current sectional area.

No special precautions were taken to avoid frictional restraint at the ends of the specimen. The increase in diameter, however, varied only slightly over the length of the specimen, except for $\frac{1}{2}$ in. at each end. After at least $\frac{1}{2}$ in. at each end had been removed, the remainder of each compression specimen was used to obtain four tension test specimens. The threaded ends of these specimens were 0.75 in. in diameter; the diameter in the reduced portion was 0.505 in. for the tests at room temperature and 0.4 in. for the tests at −188 C. From the first four compression specimens, it was possible to prepare tension test specimens that were cylindrical over a gage length of 1½ to 2 in. Since the deformation of the fifth compression specimen was less uniform, the derived tension test specimens were machined to a curve with a 2.5-in. radius, so that the smallest section was at midlength. Tension test specimens were also machined from annealed bars that had not been compressed.

For a description of the apparatus and methods used in the tension tests, reference may be made to a previous paper (11). For a description of the methods of testing at low temperatures, reference may be made to another paper (12). The tensile load was increased or decreased much more slowly than is usual in a tension test, especially during the tests in liquid air. In the approach to fracture, the stress generally did not change more than 1000 psi. in 30 to 60 sec. Determination of the load at fracture generally was quite accurate. In addition to the tests entirely at room temperature, some two-stage tests were made. In the first stage, the specimen was extended a predetermined amount at room temperature; in the second stage, it was tested to fracture in liquid air. During the interval of about an hour between the first and second stages, the specimen remained in the machine and was kept under a load of about 500 lb. In the first stage (at room temperature) the specimen was surrounded by the metal container that was to be filled later with liquid air for the second stage. The amount of plastic deformation during the first stage was determined by several calipers, the distance between the caliper points being measured by means of a traveling microscope (20).

During the tension tests at room temperature the change in diameter was measured, by means of a dial micrometer with wedge points, to an accuracy of about 0.0002 in. The measurements were continued up to the beginning of fracture.

INFLUENCE OF PRIOR PLASTIC EXTENSION ON FRACTURE UNDER TRANSVERSE TENSION

Results of longitudinal and transverse tension tests of specimens from the steel plate are shown in Fig. 1. Plastic extension of the specimens is expressed in terms of A_0/A, in which A_0 and A represent the initial and current areas of cross-section. As values of A_0/A are represented in a logarithmic scale, abscissas represent true strains. Two pairs of curves represent the flow and fracture of longitudinal and transverse specimens at room temperature. These curves have been established by numerous determinations of corresponding loads and diameters of the minimum section; the determinations were continued almost to the beginning of fracture. The curves do not connect with the points representing breaking stresses determined in the usual way, by dividing the "breaking load" by the area of cross section measured after fracture. The area so determined was less than the area at the beginning of fracture, because the metal at the rim of the cross-section continued to extend after fracture began at the axis. The breaking stresses indicated by points R, therefore, are too high. The correct fracture stress would be determined by dividing the load at the beginning of fracture by the sectional area at that instant. The points (R_T) representing the beginning of fracture are at the intersections of the flow-stress curves with the nearly straight lines extending downward from points *R*. The slope of each of these lines is such that the percentage decrease in the breaking stress is equal to the percentage decrease of A_0/A (20).

As illustrated by the pair of curves obtained with the steel in the hot-rolled condition (*BD*), the tensile properties in the longitudinal and transverse directions differed very little. The fracture stress and ductility were very slightly greater in the direction of the hot rolling than in the transverse direction. The two curves obtained with the cold-extended plate (*BDM*) do not differ greatly except at and near the yield point. The prior plastic extension of the plate evidently increased the yield point much more in the longitudinal than in the transverse direction. Moreover, the flow stress dropped abruptly at the yield of the longitudinal specimen, but not at the yield of the transverse specimen. Plastic extension of only about 3 per cent, however, raised the flow stress of the transverse specimen almost up to that of the longitudinal specimen. With further plastic extension, the percentage difference between corresponding flow stresses remained about the same.

The great difference between the yield points of the longitudinal and transverse specimens is an illustration of the anisotropic effect of plastic deformation on the elastic limit and the flow stress. Whereas plastic deformation increases the yield-stress range[4] in all directions, it shifts the range so that the mean stress of the range varies with the direction. Although the prior plastic extension of the steel plate increased the yield stress range (work-hardened the metal) in all directions, it shifted the range more in the longitudinal than in the transverse direction. If the plate had been extended the same amount by cold rolling instead of by tension, the width would not have decreased and the difference between the yield points of longitudinal and transverse specimens probably would have been less. The shift of the yield stress range in the direction of plastic extension or compression, known as the Bauschinger effect, will be discussed in the next section of the paper.

The difference between the longitudinal and transverse fracture stresses at room temperature and the difference in ductility are only slightly greater for the cold-extended plate than for the plate in the hot-rolled condition. The prior extension of the steel plate (11.3 per cent), however, decreased the ductility in both directions more than would be expected. Since the fracture stresses in both directions are a little higher for the cold-extended plate than for the plate in the hot-rolled condition, the results of the tests at room temperature give no support to the view that plastic extension lowers the fracture stress in transverse directions. However, since the prior extension of the plate was small in relation to the subsequent extension of the test specimens, more definite conclusions about the influence of plastic extension on the transverse fracture stress can be obtained from a study of the results of the tension tests at −188 C.

The results of the tension tests at −188 C. show at a glance that plastic extension of the steel plate has increased both the longitudinal and the transverse fracture stresses. The relative increases of the fracture stresses in the two directions, however, can be determined only by a detailed study of the evidence. Attention will be given first to the results of the tests entirely at −188 C., the results indicated by the small open circles and triangles. The specimens from the plate in the hot-rolled condition (*BD*) yielded before fracture. The flow stress dropped abruptly, and fracture occurred during the rise from the lower yield point, but while the stress was still below the upper yield stress. The longitudinal specimens from the cold-extended plate (*BDM*) fractured with practically no plastic deformation. The transverse specimens from this plate, however, extended slightly, with continuous rise of the flow stress.[5] Comparison of the fracture stresses obtained in the tests entirely at −188 C. shows that the prior plastic extension of the steel plate has increased the fracture stress about two thirds as much in the transverse direction as in the longitudinal direction. The mean increase of the fracture stress, however, was about five sixths of the increase of the longitudinal fracture stress. The results thus indicate that nearly all the increase in the longitudinal fracture stress, and all the increase in the transverse fracture stress, were work-hardening effects similar to the effect of plastic deformation on resistance to flow. The significance of the slight anisotropy will be revealed by a study of the results of the two-stage tests.

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[4] The "yield stress range" is defined as the range of stress between the compressive yield stress and the tensile yield stress.

The results of the two-stage tests are indicated in Fig. 1 by the small filled-in circles and triangles. In the first stage of each of these tests the specimen was extended a predetermined amount at room temperature; in the second stage, it was tested to fracture in liquid air. The curves thus obtained show the influence of plastic extension at room temperature on the fracture stress in liquid air. In the second stage, the specimens showed practically no ductility unless they had been extended more than about 50 per cent in the first stage. Similar increases in second-stage ductility have been discussed in a previous paper. With continued extension in the first stage, the second-stage ductility eventually decreases (20). In drawing the curves to represent the influence of plastic extension at room temperature on the second-stage fracture stress, allowance must be made for the influence of any plastic extension at −188 C. The curves must pass between the points representing yield and fracture (20).

A single curve in Fig. 1 represents the influence of plastic deformation at room temperature on the second-stage fracture stress of the plate in the hot-rolled condition. After an abrupt initial descent corresponding to the drop of the flow stress from the upper yield, the curve rises continuously, and is similar in form to the corresponding curve of flow stress at room temperature. The two fracture-stress curves obtained with specimens from the cold-extended plate are similar in form to the corresponding curves of flow stress. The uppermost fracture-stress curve has an initial steep descent; the fracture-stress curve obtained with transverse specimens rises continuously from the origin. The initial steep drop of the uppermost curve carries it down almost to the curve obtained with the transverse specimens. After the subsequent rise of the uppermost curve, the two curves diverge only slightly, and are relatively no farther apart than the curves of flow stress at room temperature.

The results of the experiments with the steel plate, therefore, show that plastic extension increases the fracture stress in both the longitudinal and transverse directions. They also show that the effects on the fracture stress are no more anisotropic than are the effects on the flow stress.

The evidence (Fig. 1) that plastic extension at room temperature increases the fracture stress as determined at −188 C. supports the view, expressed in previous papers (9 to 20), that plastic extension at room temperature increases the fracture stress at the same temperature. In order to convert a fracture-stress curve obtained at −188 C. to an equivalent curve representing the variation of the fracture stress at room temperature, the corresponding ordinates of the former curve should be multiplied by a constant factor, as explained in a previous paper (20). In Fig. 2, the fracture-stress curves have been so derived from the fracture-stress curves of Fig. 1, and have been adjusted to the corresponding flow-stress curves. The conversion factor used in this derivation is 0.62. Since the effect of plastic deformation on the fracture stress is a work-hardening effect, similar to the effect on the flow stress, similar pairs of curves can be used to represent the relation between the flow stress and fracture stress for any metal.

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[5] All the fractures at −188 C. were crystalline.

EFFECTS OF COMPRESSION AND EXTENSION ON THE FLOW STRESS AND FRACTURE STRESS

Figure 3 shows results of experiments made to determine how the flow stress and fracture stress are affected by compression, by extension, and by compression followed by extension. Tensile stresses are represented as positive, and compressive stresses as negative. Abscissas represent true strains determined with reference to the annealed condition. Values of A_0/A greater than 1.0 represent extension; values less than 1.0 represent compression. For example, an effective length ratio of 0.9 indicates that the equivalent length has been decreased 10 per cent. Only plastic strains are represented.

Results of Tests at Room Temperature:

Attention will be given first to the results obtained at room temperature. The small triangles on the compression curve C represent the stresses and strains at the ends of the five compression tests. From each of the five compression test specimens, four tension test specimens were prepared as described above in the section on Materials, Specimens, and Methods of Testing. One of the four tension test specimens was used for a test at room temperature; the others were used for tests at −188 C. The vertical broken lines extending upward from the small triangles on curve C represent the release of the compressive stresses. The intersections of these lines with the axis of abscissas are the origins of the tensile flow-stress curves 2 to 6. Curve 1 is a tensile flow-stress curve obtained with the steel in the annealed condition. During each of these tension tests frequent determinations were made of corresponding loads and diameters, and these determinations were continued practically to the beginning of fracture. The fractures (at room temperature) were entirely transverse. They began at the axis as dull fractures, but about half way to the periphery, they changed to partly crystalline or entirely crystalline fractures. The area of cross-section changed very little during fracture.

Comparison of curves 1 to 6 shows that the prior compression has affected the entire course of each tensile flow-stress curve from yield to fracture. As a result of the compression, curves 2, 3, 4, and 5 are tilted forward toward curve 1; the effect is most prominent in curve 2. This effect, known as the Bauschinger effect, is most prominent when the prior compression is small. Curves D represent quantitatively the variation of the slope of the tensile flow-stress curve with prior compression. Curve $D_{0.2}$ represents approximately the influence of prior compression on the tensile yield stress (offset = 0.2 per cent). During compression, the tensile yield stress first decreases, then increases. As illustrated by curve C, however, compression causes a continuous increase in the compressive flow stress. Moreover, even during the initial decrease in the tensile yield stress, there is a continuous increase in the yield-stress range. The course of the tensile yield-stress curve ($D_{0.2}$) evidently is the resultant of two effects of the prior compression. In the initial descent of the tensile yield-stress curve, the dominant influence is a downward shift of the yield-stress range; in the rise of the tensile yield-stress curve, the dominant influence is the continuous increase of the range (work-hardening). Before compression began, half the yield-stress range was in compression (Fig. 3). After the downward shift of the range, about three fourths of the range was in compression, and this distribution persisted during the further increase of the yield-stress range.

The intersections of the tensile flow stress curves with the axis of ordinates mark the ends of five strain cycles (curves 2 to 5), each of which started at the origin of coordinates. Comparison of the stresses at the ends of the strain cycles shows how the work-hardening effect increased with the size of the strain cycle. During extension after compression, the yield-stress range is shifted in the reverse direction, and eventually most of the range is in tension. Even at tensile fracture of four of the specimens (curves 2, 3, 4, and 5), however, the effects of the initial shift of the range were still apparent. Although the ductility of each of these four specimens, measured from the beginning of the tension test, was greater than that of an uncompressed specimen (curve 1), the fracture stresses were a little less. In the tension test of the specimen that had been compressed the most (curve 6), however, the greater work-hardening effect manifests itself in the higher course of the flow-stress curve and in the higher fracture stress.

Results of Tests at − 188 C.:

Two or more of the tension test specimens derived from each compression specimen were used for tests in liquid air. Tests were also made with steel in the annealed condition. A few of the tests were made entirely at −188 C., but most of them were two-stage tests. The two-stage tests were made so as to determine the influence of extension at room temperature on the fracture stress at −188 C. This relationship is represented by six curves. One of these shows the influence of extension on the fracture stress of the annealed steel; the others show the influence of extension after compression. One purpose of the tests made entirely at −188 C. was to determine the influence of compression alone on the tensile fracture stress at −188 C. Some of the specimens, however, were so ductile at −188 C. that allowance must be made for the increase of the fracture stress during plastic extension at that temperature.[6]

When the net strain at fracture in liquid air was extension or only slight compression, fracture occurred with practically no ductility. The ductility at fracture increased with the net compression, whether the test was made entirely at −188 C. or in two stages. Consequently, in drawing the curves to represent the influence of compression and extension at room temperature on the fracture stress at −188 C. it was necessary to make allowance for the influence of any plastic extension at −188 C. When the ductility at −188 C. is considerable, such allowance can be made only approximately. In making this allowance, the curves representing the results of two-stage tests have been constructed so that their origins are in proper relation to curve T_0, which represents the influence of compression alone on the tensile fracture stress at −188 C.

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[6] All the fractures in liquid air were crystalline.

As illustrated by curve T_0, compression first decreases the fracture stress to a minimum, reached at a small compressive strain, and then increases it continuously. Because of the ductility of some specimens at that temperature, the course of curve T_0 in its rise from the minimum could not be based entirely on the results obtained at −188 C. The curve has been drawn on the assumption that it should be similar in form to curves D. The evidence indicates that the course of curve T_0, like the course of curves D, is the resultant of two effects of the prior compression, namely, a shift of the yield-stress range and a work-hardening effect.

The curves based on the two-stage tests are similar in form and interrelationship to the tensile flow stress curves obtained at room temperature. The intersection of each of these curves with the axis of ordinates represents the fracture stress obtained after compression followed by an equal amount of extension. All the fracture stresses corresponding to these intersections are higher than the fracture stress obtained with steel in the annealed condition. Moreover, the larger the strain cycle, the higher is the fracture stress. Since the net strain at the end of each cycle was zero, the elevation of the fracture stress cannot be attributed to a reorientation of internal flaws, but must be viewed as a work-hardening effect, similar to the effect of plastic deformation on the flow stress. The evidence in Figs. 1 and 3, therefore, leads to the conclusion that any kind of plastic deformation of a polycrystalline metal affects the fracture stress in the same way that it affects the flow stress.

Influence of a Compression-Extension Cycle on the Fracture Stress at −188 C. and at Room Temperature:

Corresponding to the large flow-stress cycle that includes curves C and 6 in Fig. 3, there is a large cycle of fracture stress at −188 C. During the compression phase of the flow-stress cycle, the tensile fracture stress varies as indicated by curve T_0; during the tension phase of the flow-stress cycle, the fracture stress varies as indicated by the uppermost curve in Fig. 3. This fracture-stress cycle, however, represents the variation of the fracture stress at −188 C. corresponding to variation of the flow stress at room temperature. To represent the relationship between cycles of flow and fracture at room temperature, it is necessary to use the same method that was used in deriving the fracture-stress curves of Fig. 2. For this purpose, the large cycles of flow stress and fracture stress in Fig. 3 have been reproduced in Fig. 4. The curves obtained by tensile extension of annealed steel have also been reproduced.

To derive the curves to represent the variations of the fracture stress at room temperature, it is necessary to multiply by a constant factor the ordinates of the curves representing the variations of the fracture stress at −188 C. The conversion factor used in deriving curves T' from curves T in Fig. 4 is 0.69.

The form of the loop representing the influence of a compression-tension cycle on the fracture stress depends on the strain range of the cycle. With the narrowest strain range represented in Fig. 3 a loop of a different form was obtained. The fracture stress did not increase during the compression phase.

INFLUENCE OF REPETITION OF STRESS CYCLES ON THE FRACTURE STRESS

The evidence obtained about the influence of single stress cycles on the fracture stress makes it possible to consider briefly the probable influence of repetition of stress cycles.

The influence of repetition of cycles with constant stress range on the form and width of the hysteresis loop is illustrated schematically in Fig. 5(*a*). Owing to the work-hardening effect of each strain cycle, the elastic part of the stress range increases and the strain range decreases. After a certain number of cycles, the form of the corresponding loop of fracture stress would be as represented qualitatively in diagram (*a'*). (No significance should be attached to the distance between *a* and *a'*.) The width of the loop of fracture stress is equal to the strain range of the loop of flow stress. The height of the loop of fracture stress, however, is determined by the stress changes during flow at each end of the flow-stress loop.

Figure 5(*b*) represents schematically the relation between flow stress and fracture stress when the stress range is above the fatigue limit. With cyclic repetition, the curve of flow stress is elevated faster than the curve of fracture stress. When the stress range is above the fatigue limit, the curves of flow stress and fracture intersect while the width of the hysteresis loop is still decreasing. Diagram *C* represents the relationship when the stress range is about at the fatigue limit.

In order to show the curves of fracture stress in diagrams *B* and *C*, it was necessary to make some of them too long. With decrease in the width of the hysteresis loop, the loop of fracture stress would become smaller, and would become a point when the hysteresis disappears or is reduced to the condition known as elastic hysteresis.

CONCLUSIONS

Plastic extension increases the fracture stress in both longitudinal and transverse directions. The effect on the fracture stress is no more anisotropic than the effect on the flow stress.

Plastic compression decreases the tensile fracture stress to a minimum value, reached at a small amount of compression, and then increases the fracture stress continuously. The effect on the tensile fracture stress is similar to the effect on the tensile flow stress. The effect is the resultant of two factors, namely, a shift of the elastic range and a work-hardening effect.

Compression in any amount followed by an equal amount of extension, increases the tensile fracture stress. The effect on the fracture stress increases with the strain range.

The effect of plastic deformation on the fracture stress cannot be attributed to reorientation of internal flaws.

Any kind of plastic deformation of a polycrystalline metal or alloy affects the fracture stress in the same way that it affects the corresponding flow stress.

The results of these tests confirm the conclusion expressed in previous papers (9 to 20), that the influence of plastic extension on the flow stress and fracture stress can be represented by two curves rising and intersecting at a small angle. The ductility thus is sensitive to the differential effect of any variable on the flow stress and fracture stress.

Too much emphasis has been placed on the hypothesis of internal flaws in attempts to account for the conditions affecting fracture of metals.

Since the fracture stress and flow stress are similarly affected by plastic deformation and by the stress system, and are similar functions of temperature and the strain rate, the mechanism of fracture possibly is closely related to the mechanism of flow.

Acknowledgments:

Acknowledgment is due to Miss Frances J. Cromwell for assistance in the experiments and for preparing the illustrations, and to D. H. Woodard for occasional assistance with the experiments.

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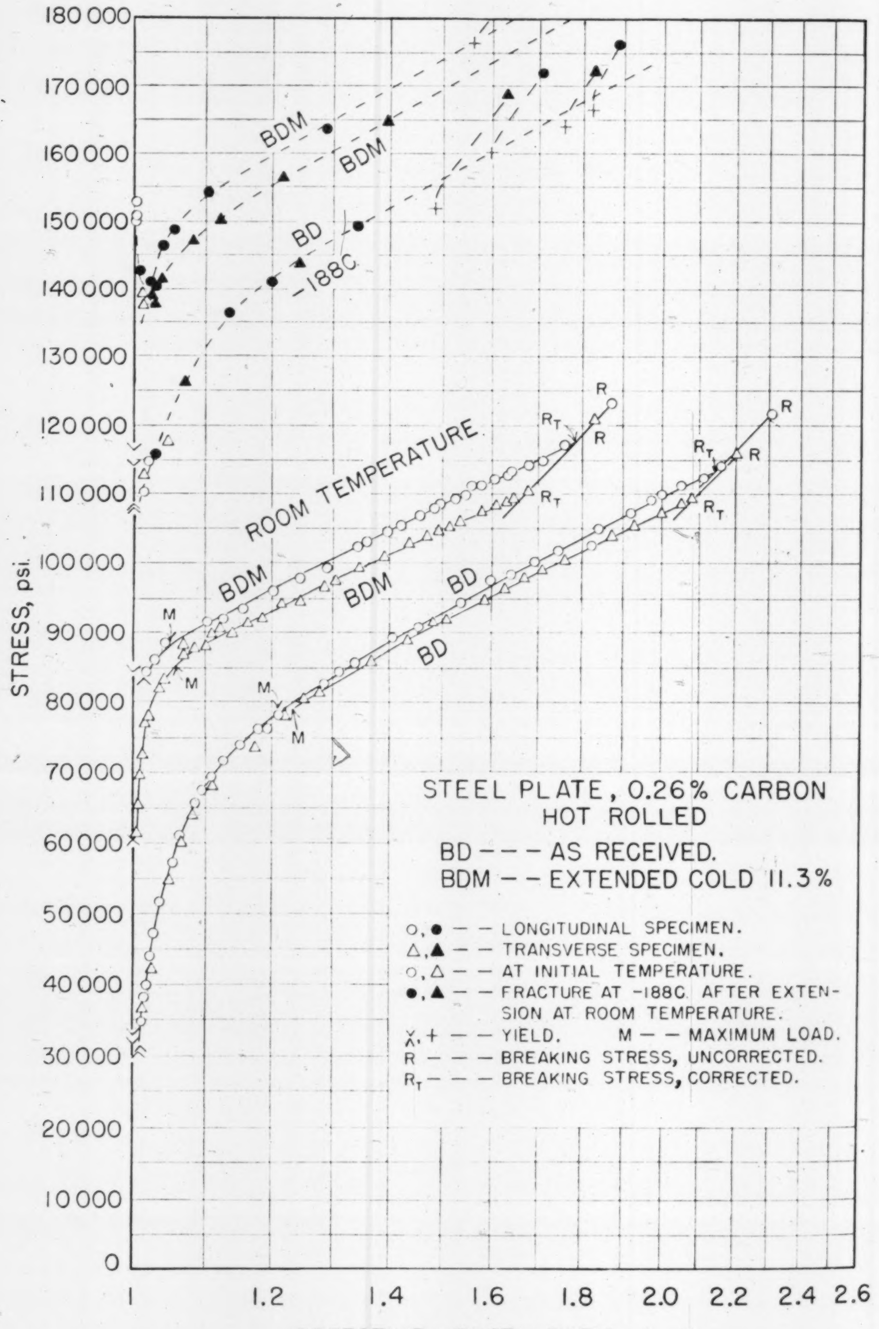

Fig. 1.—Influence of Prior Plastic Extension on Flow and Fracture Under Transverse Tension.

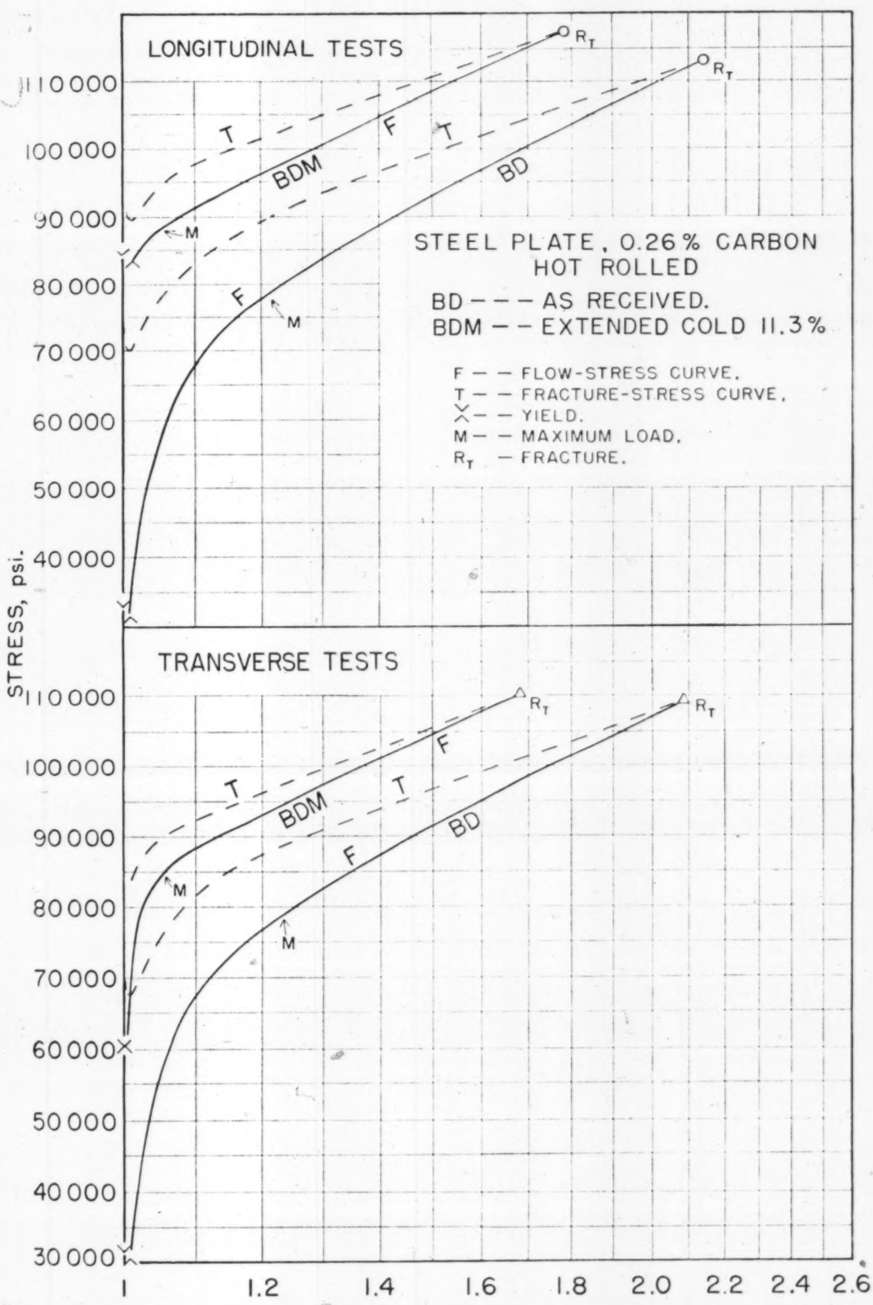

Fig. 2.—Influence of Plastic Extension on the Flow Stress and Fracture Stress at Room Temperature.

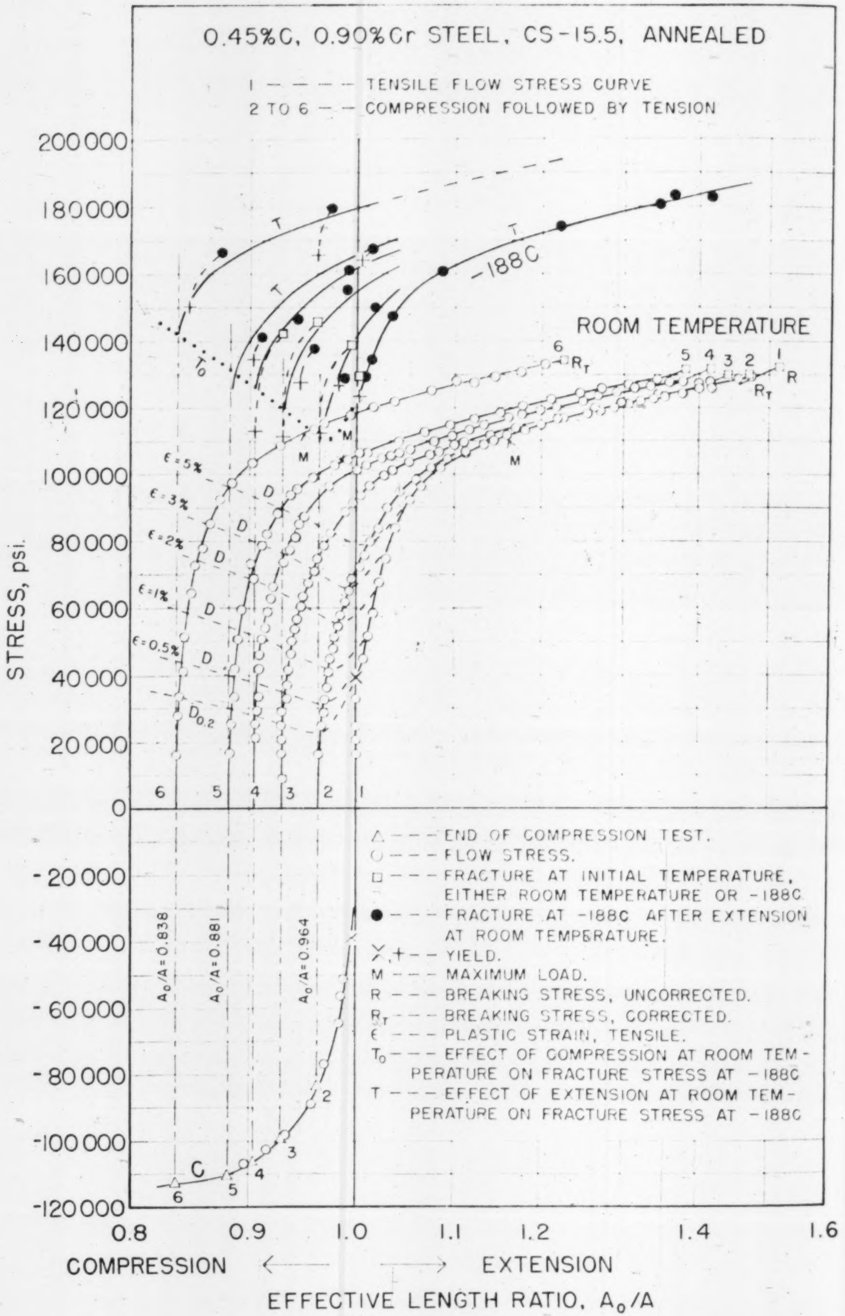

FIG. 3.—Influence of Compression and Extension on the Tensile Flow Stress and Fracture Stress.

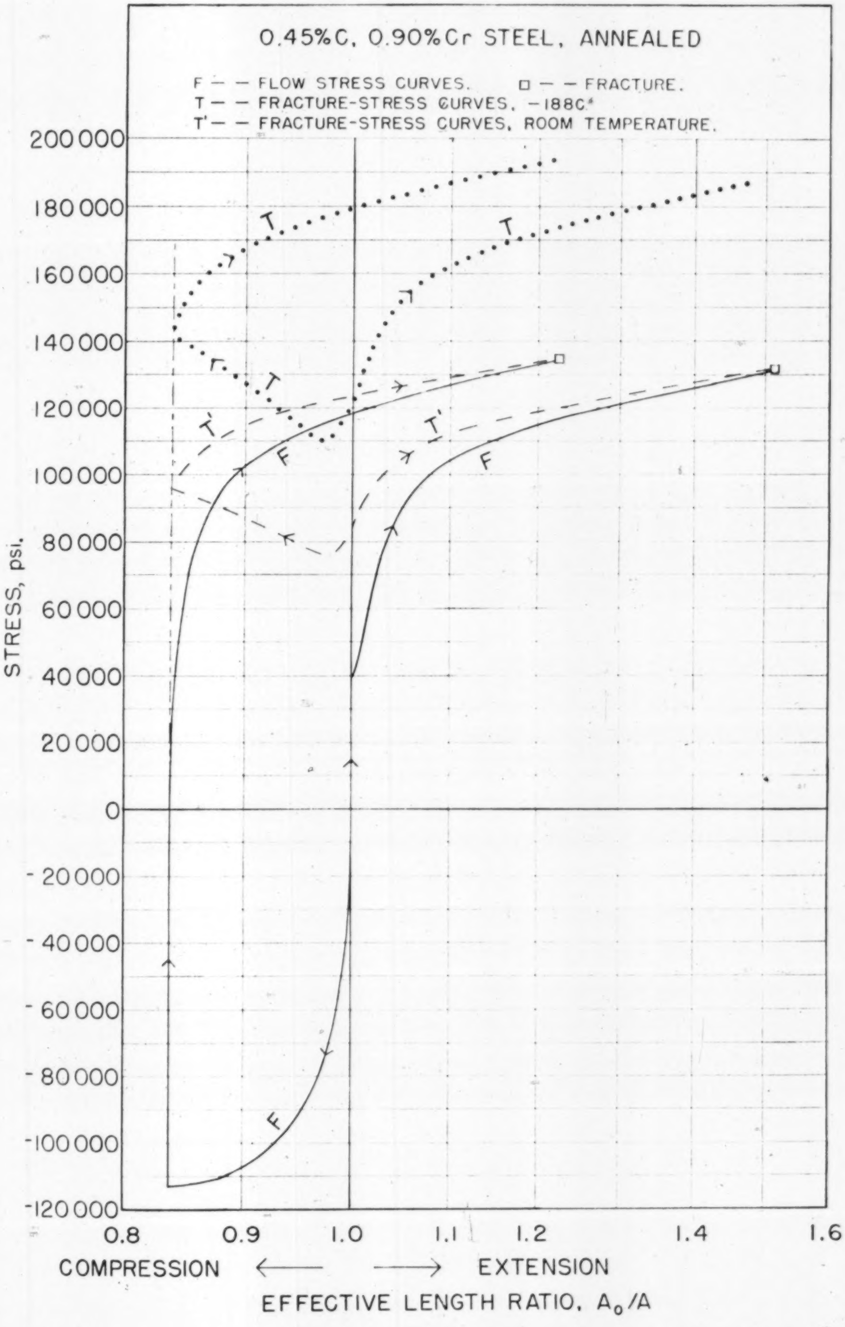

Fig. 4.—Variation of the Flow Stress and Fracture Stress with Extension and with Compression Followed by Extension.

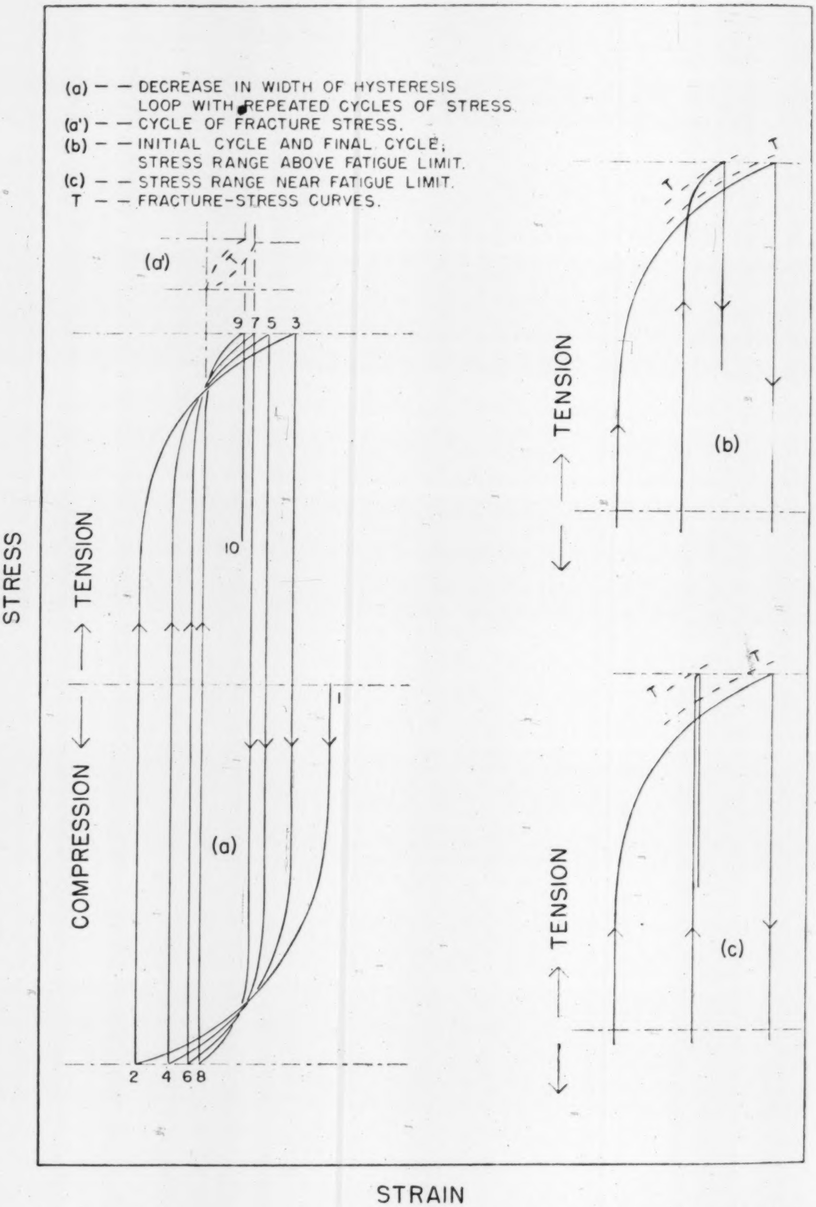
(a) — — DECREASE IN WIDTH OF HYSTERESIS LOOP WITH REPEATED CYCLES OF STRESS.
(a') — — CYCLE OF FRACTURE STRESS.
(b) — — INITIAL CYCLE AND FINAL CYCLE; STRESS RANGE ABOVE FATIGUE LIMIT.
(c) — — STRESS RANGE NEAR FATIGUE LIMIT.
T — — FRACTURE-STRESS CURVES.
(a')
T
9 7 5 3
10
1
(a)
2 4 6 8
STRESS
COMPRESSION ⟵ | ⟶ TENSION
⟵ | ⟶ TENSION
(b)
T
T
⟵ | ⟶ TENSION
(c)
T
T
STRAIN

Fig. 5.—Effect of Repeated Stress Cycles on the Flow Stress and Fracture Stress.

DISCUSSION

MR. R. A. FLINN.[1]—In my opinion, the metallurgical profession will be more grateful to Mr. McAdam and his co-workers for this paper, than for any previous contribution. All have been outstanding contributions, but the distinguishing characteristic of this paper is the care with which the authors outline their point of view. I think that the concession at the very beginning of the paper, in sacrificing their own previous terminology for that of what might be called the opposition, that is, in using fracture stress in place of the more accurate but less digestible technical cocohesive limit, is typical of the spirit of the paper. Research workers in the field of mechanical metallurgy have been able, of course, to assimilate McAdams work, but the large group of metallurgists for whom it has important implications, have not.

Recently, more spectacular presentations and, this paper indicates perhaps less cautious presentations, have awakened metallurgists to the importance of the many past works of the present authors, and that is why it is good to have this direct rebuttal of some of the work that has been published recently.

Now, to add some actual observations to these remarks which I hope have not been too general, some of the metallurgical implications of the discussion are quite important to us. For example, the question of fracture stress being almost a constant of the material, has been bothering a great many metallurgists, as though this critical stress were just lurking at a small amount above the flow stress and ready, at the slightest encouragement, to give cleavage that would cause premature failure. This paper shows that to the contrary, the mechanism of failure during fracture is similar to that causing flow, and I wish we could draw out the authors a little more on that point. One indication was their careful observation of the actual failure, of measuring the area just before failure and then area at failure. Some of the previous work has taken them both as synonymous.

Now, we were a little startled at one of the early statements in the paper that cleavage fractures, that is, where you see a bright surface, are not synonymous with brittle fractures. We have more or less taken for granted that if we had a cleavage fracture we had a brittle fracture, but to go back to one of the most ductile fractures, for example, the fracture of a zinc single crystal where you get 200 or 300 per cent elongation we remember it as a smooth cleavage fracture. A good deal of our thinking on these bright fractures has been, I think where we had grain boundary conditions. For example, during the war we made a heat of tank armor that showed grains as large as your thumb and very low ductility, with what might have been called a cleavage fracture but we believe that a grain boundary constituent was the root of the trouble and not the mechanism of fracture of the matrix material. Another example is in cast iron. We obtain a rough dull fracture with one type of graphite distribution (interdendritic) and a bright cleavage fracture with a random distribution. On the other hand, we observe the greatest ductility with the brighter (cleavage appearing) fracture.

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MR. D. J. McADAM, JR. (*author's closure*).—The authors appreciate Mr. Flinn's remarks. I would say though that I think the contrast he makes between the clarity of this paper and the preceding papers is partly due to the fact that in this paper we are dealing with only one stress system. In the preceding papers we have dealt with a combination of plastic deformation, temperature, the stress system and, in some cases, the strain rate. However, the illustrations in those papers show clearly what the coordinates are and we feel sure that anyone who will take the time to study the evidence in the diagrams, will have no difficulty in understanding the papers. Mr. Flinn has emphasized the need for further information about the relation between flow and fracture. A discussion of this subject is given in a paper on "Flow, Fracture, and Ductility of Metals," published in *Metals Technology*.[2]

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INTERCRYSTALLINE COHESION AND THE STRESS-RUPTURE TEST*[1]

By H. H. Bleakney[2]

Synopsis

In this discussion the author has tried to collect the evidence bearing on the causes of intercrystalline failure of metals when stressed at elevated temperatures. Existing theories of intercrystalline cracking have been stated, and criticized in the light of the accumulated evidence.

The evidence has been summarized and certain questions which appear to be inherent have been listed. An hypothesis has been suggested for certain kinds of intercrystalline failures; and this hypothesis has been used as a foundation upon which to construct a program of investigation to seek answers to the questions mentioned above.

Prior Work

The following quotation from Houdrement (**1**)[3] gives a brief history of the development of creep testing, and the passage in italics describes the phenomenon which is the subject of this discussion.

"The first test for steels which were to be used at elevated temperatures was the hot tensile test. The time of testing was soon increased from about three minutes to from twenty to thirty minutes. It was quickly recognized, however, that the results so obtained did not correspond to service performance. The designers, therefore, adopted as criteria, those stresses under which no inadmissible extension of the steel occurred after exposure for long periods to the temperatures employed, *i.e.*, in practice there was no significant distortion of the material. These tests required thousands of hours, and as a consequence various short expedients were adopted. Using, as a basis, the rate of extension of various steels, for which the not very happy term 'Daverstandfestigkeit' was coined, very rapid progress was made. Systematic researches were made on the action of alloying elements, and specifically on the differences in occurrence of recrystallization and lattice distortion effected by special carbides.

"A selection of steels for duty at high temperatures on the basis of limiting creep strength (daverstandfestigkeit) seemed the more justifiable as it was possible to determine that other properties, such as the fatigue strength at high temperatures, usually played no important rôle. This might be due to a decrease in notch sensitivity of steels with rising temperature under oscillating loads, but in any case, the creep strength of steels at high temperatures lies below the fatigue strength, and consequently is the controlling factor in design.

"This kind of investigation of

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*Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] Permission to publish this paper has been granted by the National Research Council of Canada.

[2] Associate Research Engineer, National Research Council of Canada, Ottawa, Ont., Canada.

[3] The boldface numbers in parentheses refer to the references appended to this paper, see p. 588.

structural materials and the grades of steel developed by this method of testing had no sooner been adopted when a new phenomenon appeared. Although the steels displayed in service a rate of extension no greater than that found in testing; *yet they broke with a brittle fracture, after relatively small total elongation and negligible reduction of area.*"

The essential mechanics of creep may be regarded as a product of the opposing influences of work-hardening produced by plastic deformation of material under an imposed load, and the softening by strain-relief under the influence of the temperature employed. So long as creep continues, the elongation of the material should be accompanied by a proportionate reduction of area. The consequent stress intensification, therefore, should eventually result in a value of the stress equal to the strength of the material, and at that point the material should immediately fail with a degree of local deformation typical of its reaction in a short-time tension test. By this reasoning, lower loads would require greater uniform reduction of area before stress intensification reached a value equal to the strength of the material, and therefore total elongation would be proportionately greater.

In practice, however, the total elongation measured after fracture occurring as a result of the application of low loads frequently proves to be less than that found after fracture resulting from the application of higher loads. Moreover, the path of fracture found in these cases is usually intercrystalline; rather than the transcrystalline path characteristic of ductile metals. This anomalous behavior compels us to recognize the existence of some influence or influences not contained in the mechanism postulated above.

In 1939, Gillett (**2**) devoted a large part of his Howe Memorial Lecture to a discussion of these influences. His conclusions may be summarized in the statement that none of the theories so far proposed is supported by all the evidence, and many more data are required before an adequate explanation can be expected. Many other writers have mentioned the limitations of our understanding of creep phenomena, and Sir Harold Carpenter and J. M. Robertson (**3**) state that "The general relations between time, temperature and deformation, constitute the most complicated subject within the scope of this book." Hoyt, discussing a paper by Parker (**4**), said "The problem of grain boundaries and intercrystalline cohesion remains very much of a mystery, though it is over thirty years since Rosenhain proposed his amorphous cement hypothesis."

A theory of intercrystalline fracture of metals grew out of Jeffries (**5**) equicohesive temperature concept, and upon that theory Gillett (**2**) trained his heaviest artillery. The theory was explicitly predicated on the validity of Rosenhain's (**11**) amorphous cement hypothesis, and with the crumbling of that foundation, one might have expected the superstructure to fall as well. However, it has received wide credence, and the tenacity with which it is retained is a real obstacle to progress toward elucidation of the causes of intercrystalline failure. As recently as 1944, Parker (**4**) attributed intercrystalline failure to relative weakness of interatomic cohesion at the grain boundaries at temperatures above the "equicohesive temperature."

Prior to the war, this problem was regarded as being largely academic. Limiting creep stresses and hence rates were so low that, with very rare exceptions, structural components were replaced after many thousands of hours of service because of slight allowable total deformation, long before fracture would have occurred. High stresses in turbosupercharger buckets and gas turbine blades, however, have given practical significance to the problem, which may prove to be a significant factor in the development of alloys for such service. The following quotation from Zener and Hollomon (**6**) indicates the changing attitude of scientific workers. "A knowledge of the plastic and rupture properties of metals is of utmost importance in many engineering applications, yet these properties have apparently not received their fullest fundamental consideration by metallurgists and physicists." Before the war, workers in the field of creep were preoccupied almost entirely with limiting creep stresses and the second stage of creep. The third stage of creep, including fracture, was generally looked upon as something to be avoided, rather than studied. In recent times, however, the work being done on metals for high-temperature service indicates that increasing importance is being attached to the third stage of creep.

When the limiting creep strength is the criterion employed, the third stage of creep has very minor significance; but when stress-rupture is the criterion, third stage creep may be the most important factor. So far as we can see today, a study of third stage creep becomes mainly a study of intercrystalline fracture. In stress-rupture tests of many alloys, the third stage of creep is found to be more protracted than the second. Hence the endeavor to increase the time to fracture with a given load and temperature becomes a problem of preventing fracture before the whole inherent ductility of the material has been utilized, and the fracture stress has increased to a value equal to the strength of the material. Prevention of premature failure requires discovery of the causes of intercrystalline fracture, which have baffled metallurgists for so many years; and subsequently, the development of methods to eliminate those causes.

So far, this discussion has emphasized the complexity of the problem, the conflicting opinions which it has evoked, and the fact that no really satisfactory explanation has been offered. It is not to be expected, therefore, that the true causative forces will be readily ascertained. On the other hand, abandonment of the equicohesive temperature concept should free research from the blind alley into which that theory leads it, and should stimulate exploration of more promising avenues.

Investigation of the causes of intercrystalline cracking under creep conditions appears to require, quite naturally, a program such as the following:

1. Accumulation of representative data from as many sources as possible including both laboratory tests and service observations.
2. Critical appraisal of existing theories in the light of the accumulated evidence.
3. Formulation of an hypothesis in harmony with the evidence.
4. Experimentation to test the validity of the hypothesis.

Reasonably thorough study of the published work on the intercrystalline failure of metals under stress at elevated temperatures does not reveal much useful quantitative data. The surprise with which one recognizes this fact rapidly diminishes, however, when one considers the very recent advent of creep testing as a metallurgical tool. It was not until 1933 that the A.S.T.M. proposed even a tentative procedure for creep testing. This was revised in 1934, 1935, 1938, and not until 1941 was it adopted as a recommended practice, after further revision. Even then, the following foreword was included:

> "Since the characteristics which render a material resistant to extension under the conditions of test are not yet completely known, the details of the procedure considered herein are those known to have important influences. Since others, equally important, may in the future be discovered, the recommended details of the method are largely advisory in nature."

Since the intercrystalline type of failure has been widely regarded as an academic, if deplorable, by-product of creep, the paucity of quantitative data is not really astonishing. Because of this lack of data, tabulation of results is not feasible, and the evidence presented will have to be confined to summarization of published works, and quotations from the opinions of the authors.

THE COLLECTED EVIDENCE OF INTERCRYSTALLINE CRACKING

Greenwood (**7a-n**) and his associates from 1932 to 1942 conducted a most exhaustive series of investigations on the creep of lead and lead alloys. Moore and his co-workers (**8a, b, c**) over nearly the same period, thoroughly investigated the creep and fatigue properties of lead alloys for cable sheaths. From these works, some interesting features of intercrystalline failure appear. Greenwood (**7c**) places intercrystalline failure of metals under stress at elevated temperatures in the same category as intercrystalline failure by stress corrosion, but he does not actually commit himself to the proposition that these phenomena are manifestations of identical causes. The following quotation from the same work reflects his opinion:

"An important question to settle is whether prolonged steady stress in the absence of a corrosive medium (other than the atmosphere) and in the absence of vibration will cause lead to fracture in an intercrystalline manner."

It is not clear why Greenwood makes an exception of the atmosphere as a corrosive medium. He says also (**7c,** p. 487) "It is, of course, well known that a fluctuating stress will cause intercrystalline fracture in lead, and it is further known that certain corrosive agents, with or without the application of stress will cause intercrystalline weakness." Previously, (**7c,** p. 478) however, he quoted Gough and Sopwith (**9**) to show that fatigue tests of lead *in vacuo* not only gave higher endurance values than fatigue tests in air, but resulted in transcrystalline rather than intercrystalline failure. Gough and Sopwith were actually much less definite than this, but examination of plate III of their paper will leave little doubt that exclusion of the atmosphere has vastly reduced, if it has not completely eliminated, the occurrence of intercrystalline failure. Hence, if exclusion of the atmosphere has so markedly reduced intercrystalline failure under the adverse conditions of alternating stress, it would seem to be imperative that similar investigation be made under the less dangerous conditions of steady stress.

Moore, Betty and Dollins (**8c**), as a result of many years' work, conclude that intercrystalline failure of lead and lead alloys under steady stress conditions is a rare and insignificant occurrence. Beckinsdale and Waterhouse (**10**) came to the same conclusion after a thorough investigation into the causes of intercrystalline cracking of lead cable sheathing in service. They found that in every case failure was attributable to fatigue. On the other hand, Greenwood (**7c**) objected that Moore, Betty, and Dollins' results were obtained from experiments involving too high stresses and too short times. In Figs. 5 to 9 of his paper Greenwood illustrates the progress of intercrystalline failure in a sample of commercial lead which broke after 470 days under a stress of 500 psi. This sample had been reheated to 130 C. for 18 hr., after rolling, and tested two days later. Three other test specimens, taken from the same material aged 25, 71, and 188 days after rolling without any reheating, and subjected to the same stress, failed without indications of intercrystalline cracking. It is a fair inference, therefore, that the type of failure was influenced by the structural condition of the material, which is controlled by composition and heat treatment. In the same paper Greenwood says "It is concluded that lead can fail by intercrystalline cracking by the application of a steady stress in air." Nevertheless, examination of the comprehensive work given in reference (**7**) forces one to the conclusion that the occurrence of intercrystalline cracking in lead is dependent upon the presence of impurities such as antimony, tin, and silver. Tellurium is especially harmful in this connection. Nowhere in the voluminous reports on the work mentioned can one find direct evidence of intercrystalline cracking in vacuum melted electrolytic lead.

Parker and Riisness (**30**) report intercrystalline failure of 99.988 copper tested at 150 C. Martin and Parker (**32**) conducted stress-rupture tests on various coppers from 99.999+ pure to 99.920, the latter estimated to contain 0.035 per cent oxygen. They state that, in general, fractures were transcrystalline for short-time tests and intercrystalline for long-time tests. The data for reduction of area, presented in Table 2 of their paper, indicate that the nature of the fracture was not noticeably influenced by the impurity content, at least in the tests reported.

H. V. Kinsey (**33**), at the Bureau of Mines (Canada) conducted stress-rupture tests on 99.95 copper which may fairly be compared with the samples employed by Martin and Parker, with respect to the type of fracture displayed. Three specimens of this material, having an average grain diameter of about 0.05 mm., and a Brinell hardness of 43, tested at 260, 245, and 230 C., under a stress of 10,000 psi., failed in 81, 165, and 252 hr., respectively. In each case, the fracture was intercrystalline, without any "necking" of the test bar.

The results presented above, and the absence of conflicting evidence, indicate that copper is highly susceptible to intercrystalline failure under steady load at elevated temperatures.

Price (**12**) has shown that short-time tension tests of copper reveal increasing ductility with increasing temperature of testing, except for a slight diminution over the temperature range of normal recrystallization. Carpenter and Robertson (**3**, p. 178) attribute this to strain aging, but it is nowhere suggested that intercrystalline failure can be obtained in short-time tension tests at temperatures under the solidus.

Very well informed sources were asked whether they could cite references to cases of intercrystalline failure in light alloys at elevated temperatures. They replied that they did not know of any examples of this type of failure in the materials specified. Hanson (**31**) mentions intercrystalline failure of aluminum, but his comments are not properly applicable to this discussion.

The Timken Roller Bearing Co. is one of the leading organizations engaged in the development and production of heat-resisting steels. Over a period of years they have sponsored researches on creep at the University of Michigan. In 1938 A. E. White, C. L. Clark, and R. L. Wilson (**13**) published results of these researches involving rupture and intercrystalline failure of steels which fairly represent the known facts at that time. The most significant features of this work are set forth in Fig. 1.

The curve for the chromium-molybdenum-silicon alloy is representative of material having good creep strength but inferior oxidation resistance. The curve for the 5 per cent chromium-molybdenum-silicon alloy represents a material with lower creep strength but good oxidation resistance at the temperature shown. The curve for S.A.E. 1015 steel represents material inferior with respect to both creep strength and oxidation resistance. There are two very significant features of these curves. The first is the discovery by the authors of the power function relationship between the originally applied stress and the time to fracture. The second, and most important for our purposes, is the sharp break in the curves of the materials having inferior oxidation resistance. General and uniform oxidation would be expected to affect the slope of the curve right from the beginning, but the sharp break suggests the intrusion of a new factor under critical conditions. This feature will be examined again in a later part of the discussion. However, the data indicate that the break in the curve is associated with intercrystalline fracture, which, in turn, is associated with oxidation resistance. The authors specifically state that "In general the statement is correct that the time required for the break to occur in the logarithmic relationship is proportional to the oxidation resistance, and this is especially so at the more elevated temperatures."

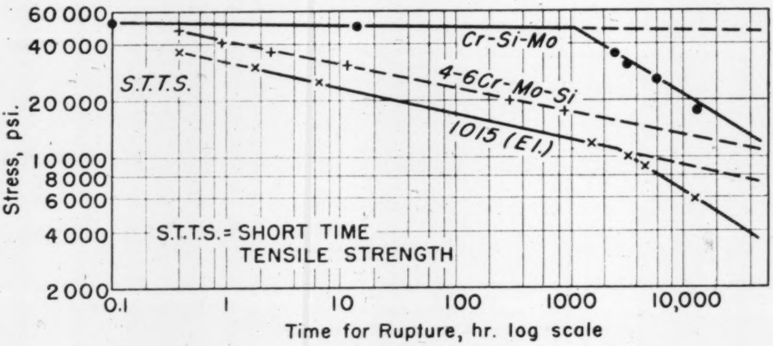

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FIG. 1.—Stress-Rupture Curves of Steels Shown.

Thielemann and Parker (**14**) obtained confirmation of the logarithmic relationship between applied stress and time, and also found sharp breaks in the curves of carbon steels. They also showed that ingot iron tested in hydrogen did not display the break in the curve; and when the same material was tested after painting with aluminum paint, the break was less pronounced. However, they obtained intergranular fractures in all cases, and concluded (page 570) that "Results from tests on these three materials indicate that intergranular failures are inherent to certain steels and cannot be attributed to intergranular oxidation." They are quite right in this particular case, as the ingot iron they employed contained 0.037 per cent sulfur and only 0.005 per cent manganese; and the presence of the resulting iron sulfide at the boundaries would vitiate any conclusions as to the influence of oxidation on intergranular failure (**29**). Jenkins and Mellor (**15**) tested mild steel and iron *in vacuo* for relatively short times and found the same intergranular failure in the case of ingot iron having a similarly dangerous sulfur-manganese ratio. The other materials, with higher manganese, were all free from intercrystalline cracks, but that is not significant as they would have displayed normal ductility if tested in air for the same short time. 18 per cent chromium, 8 per cent nickel corrosion-resistant steel is notorious for intergranular failure when stressed at high temperatures, but that behavior is attributable to the diffusion of carbon to the grain boundaries, which it depletes in chromium by the formation of chromium carbide. The evidence appears to warrant the conclusion that, except for abnormal materials such as ingot iron and 18-8 corrosion-resistant steel atmospheric oxygen controls the incidence of intercrystalline failure, and the break in the stress-time curve.

Some further evidence in this connection is contained in the work of Agnew, Hawkins, and Solberg (**16**). In Table 5 of their paper, they showed the ratio of stress in steam to stress in air required for rupture of various steels in 10,000 hr. at 1200 F. Low-carbon steel and 12 per cent chromium steel had over three times the value in steam that they had in air; and carbon-molybdenum steel had a ratio of 1.8 to 1. The other alloys involved in the tests were not greatly affected. Accurate comparison is not possible, but qualitatively the order of improvement in stress-rupture strength obtained by testing in steam rather than air is about the order of susceptibility to corrosion in the two media. The most rigid sceptic would be hard put to pass over these indications of the great influence which the atmosphere exerts upon the rupture strength of materials.

THEORIES OF INTERCRYSTALLINE RUPTURE

Jeffries' equicohesive temperature theory has already been mentioned. Rosenhain's amorphous metal hypothesis (**11**) formed its foundation; and the validity of that hypothesis was explicitly asserted, although Desch and Smith, in discussing Rosenhain's paper (**11**), had asked some pertinent questions which were never satisfactorily answered. Rosenhain had found that many metals broke with an intercrystalline fracture when tested in tension at high temperatures. Jeffries (**17**) experimented with coarse and fine-grained iron, tungsten, silver, gold, and platinum, and found that for each metal there was a certain temperature above which the coarse-grained samples were stronger, that is, more creep-resistant, than the fine-grained, and below which the fine-grained material was stronger than the coarse. He attributed this phenomenon to the viscosity of the "amorphous metal" at the grain boundaries, and postulated that the viscosity of the grain boundary material was greater than the resistance to slip of the crystalline grains, below a certain temperature. Hence the grain boundaries were stronger than the grains. This "amorphous metal" was supposed to lose viscosity faster than the crystalline metal lost slip resistance as the temperature was raised, so that for each metal there was a temperature above which the grain boundary material was weaker than the grains themselves. The temperature at which the grain boundaries and the grains were of equal strength was called the equicohesive temperature. This concept quite happily explained the response to rising temperature of coarse- and fine-grained metal under stress. It also explained intercrystalline failure at high temperatures. The fact that this temperature was also the temperature at which the metal recrystallized under the conditions employed was treated as a coincidence.

At the September, 1918, meeting of the A.I.M.E., Merica (**18**) advanced objections to this theory both theoretical and practical. These were not completely met, but the concensus of metallurgists appears to have been that the theory was an enchanting solution to a nasty problem and the objections were generally overlooked. This theory, therefore, was accepted, apparently on the ground proposed later by Chipman (**19**) that the real merit of a theory depended upon whether it got you out of more trouble than it got you into.

The virtual, if not complete, abandonment of the amorphous metal hypothesis has cut most of the ground out from under the equicohesive temperature theory. However, there must be considerable atomic disorder at grain boundaries, and the theory had become so firmly settled that the same reasoning was continued on this new and rather flimsy foundation. Gillett (**2**) presented a mass of evidence to show that this theory does not contribute to the search for an explanation of intercrystalline failure, and dismissed it with these words "Giving a name to something we do not know about does not add to our knowledge."

With respect to the coarse *versus* fine-grained metal behavior, it is true that coarse-grained material appears to have better creep resistance than fine-grained. This is not invariably so, however, and in many cases the difference noted is too slight to be significant. There are so many variables which enter into creep phenomena that it would not be unfair to infer that the apparent effect of grain size may not be real, but may be due to another variable, such as precipitation hardening, which occurs as a result of the treatment which produced the difference in grain size.

The picture of intercrystalline failure as a result of viscous flow at grain boundaries is equally unsatisfactory. It is not denied that grain boundary motion is possible; indeed grain rotation has been demonstrated, (**8c**), but it is not safe to infer that such motion involves fracture. Such motion need merely involve a redistribution of atoms; and in so far as the disordered atoms may be considered amorphous, they would be incapable of the strain-hardening hitherto regarded as prerequisite to fracture of ductile metals under unidirectional stress. In the final analysis, the onus is on the proponents of the theory to explain why copper fails with intercrystalline fracture after 81 hr. or less, while lead exhibits fully ductile failure after more than 400 days. If it cannot be said that this theory is not completely disproved, it can at least be said that it has not been proved and, again quoting Gillett (**2**) "I cannot see that the concept of an 'equicohesive temperature' as applied to creep is of the slightest service in the practical solution of creep problems, or in clarifying our conceptions of the phenomena involved."

Parker (**4**) proposed a theory of intercrystalline failure involving an assumption of imperfections in the grain-boundary material, such as "voids where a few atoms are missing from the structure." Aside from the difficulty of reconciling this concept with the laws of thermodynamics, the whole theory is based on the validity of Jeffries' equicohesive temperature theory and can carry no more weight than that theory will sustain.

Carpenter and Robertson (**3**, pp. 193, 194), discussing the effect of time, temperature, and deformation upon metals employ an ingenious explanation for intercrystalline failure. They observe that recrystallization involves continuously changing grain boundaries, and therefore promotes mobility of atoms at the changing boundaries. They believe that an increased rate of extension in the direction of the stress occurs as a result of the increased atomic mobility. They also state that "In the course of the adjustment cohesion at the moving boundaries is easily ruptured and intercrystalline fracture initiated."

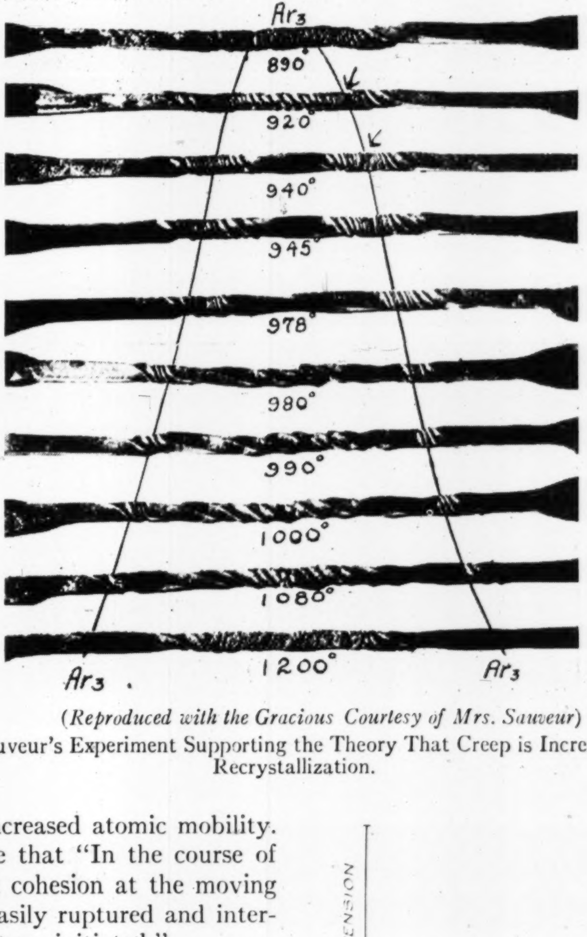

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FIG. 2.—Sauveur's Experiment Supporting the Theory That Creep is Increased During Recrystallization.

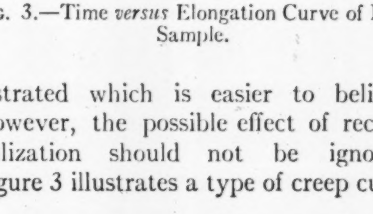

FIG. 3.—Time *versus* Elongation Curve of Lead Sample.

The suggestion that rate of extension, that is, creep, is increased during recrystallization is supported by some experimental verification. Figure 2 illustrates an experiment devised by Sauveur (**20**). It is evident that the metal is exceptionally plastic at the regions where recrystallization is taking place, and it could be inferred that the increased plasticity is contributed by the recrystallization. The fact that gamma iron is less plastic than alpha iron at nearly equal temperatures is an explanation of the phenomenon illustrated which is easier to believe. However, the possible effect of recrystallization should not be ignored. Figure 3 illustrates a type of creep curve which Greenwood obtained from tests of lead (**7b**, p. 97). Greenwood appears to believe that the increased rate of extension at the beginning of the third stage of creep is interrupted by recrystallization, but the author finds it easier to believe Carpenter and Robertson's explanation. From their reasoning it would be inferred that the sample illustrated by the curve had not entered the third stage of creep until after the hump was passed. Instead, the hump in the curve would be explained by the increased flow rate permitted by recrystallization. If this is so, their theory receives confirmation, and added significance attaches to Sauveur's experiment. Hanson (**31**) presented well documented arguments to support a definite assertion that the creep rate is greatly increased during recrystallization.

The use of the mobile boundary concept in connection with intercrystalline failure is much less acceptable. Singleton (**21**) attributed intercrystalline failure of lead to this alleged cause, but his contention was convincingly refuted by Greenwood (**7c**, p. 489) as follows: "If the lead is recrystallizing, and the position of the boundaries changing, then it is difficult to imagine where cracks would form. On the other hand, if the lead is in a condition to resist recrystallization, then the applied stress constantly acts on the same boundaries, and if any weakness is associated with these they would tend to crack." He goes on to confirm this contention by reporting that of four samples which differed only in heat treatment, the only one which displayed an intercrystalline fracture was the only one which had not recrystallized during the test. The evidence, therefore, is consistent with Carpenter and Robertson's belief that during recrystallization under stress, the creep rate is accelerated. On the other hand, the evidence is not only inconsistent with their proposed mechanism of intercrystalline failure; it is directly contrary to that theory.

SUMMARY OF THE EVIDENCE AND REAPPRAISAL OF THE PROBLEM

The three theories outlined above are the only ones found which deal with intercrystalline failure; and none of them appears to be useful, even as a working hypothesis. Most of the data from which to deduce such a theory have been obtained as a by-product of creep and stress-rupture investigations, and they are simply not adequate. The solution of the problem quite clearly requires further experimentation, devised specifically to obtain data on the nature of the forces which promote intercrystalline failure. The following is an attempt to formulate the questions to which answers are required and to suggest methods of obtaining such answers.

It is first necessary, however, to summarize and examine the evidence as follows:

1. Pure lead fails with a normal ductile fracture, free from intercrystalline cracking, when subjected to a steady stress in air.
2. Impure lead (about 0.01 per cent total impurities) under relatively low stress in air, fails with markedly reduced ductility and intercrystalline fracture. Susceptibility to intercrystalline failure is increased with increasing impurity.
3. Intercrystalline cracking of lead is influenced by heat treatment. Greenwood (**7c**) advanced arguments to support a contention that grain-boundary precipitation of impurities promotes intercrystalline cracking.
4. Lead exposed to vibrating stress in air fails inevitably by intercrystalline cracking. The same stresses *in vacuo* result in failures free from intercrystalline cracking.
5. Copper under prolonged load at

temperatures over 200 C. in air is very susceptible to intercrystalline cracking.

6. A logarithmic relationship exists between stress and time to failure when the test conditions involve relatively slight oxidation. As conditions change in the direction of increased oxidation, intercrystalline cracking appears and failure occurs much sooner than would be anticipated from extrapolation of the logarithmic curve. Increasing oxidation causes the break in the curve to occur at decreasing time intervals.

7. Prima facie evidence indicates that certain materials, for example, iron with a high sulfur-manganese ratio, fail by intercrystalline cracking because of weakness conferred by the intercrystalline material. Other materials, notably 18-8 corrosion-resistant steel, fail in this way because precipitation of a second phase at the grain boundaries is accompanied by lowered oxidation resistance of the adjacent material.

What is the significance of these data, and what questions do they suggest that require answers? No doubt different questions would occur to different investigators, but the author believes that the following are obvious and would present themselves to all students of the subject:

1. Why does lead break with a fully ductile fracture after a test of 10,000-hr. duration; while copper has been found to break with a brittle intercrystalline fracture after only 81 hr. or less?

2. What is the mechanism by which very small quantities of impurities change the nature of the fracture in lead from ductile transcrystalline to brittle intercrystalline?

3. Why do vibrating stresses superinduce intercrystalline fractures in lead when tested in the atmosphere; but result in transcrystalline fractures when the tests are conducted *in vacuo*?

4. What is the nature of the factor which appears in stress-rupture tests of metals, to alter the logarithmic relationship between stress and time to failure?

5. Is there any relationship between the forces which germinate intercrystalline cracking of metals under stress at elevated temperatures and those which result in intercrystalline failure through the processes of stress-corrosion?

6. What influence is exerted by precipitation phenomena upon intercrystalline cracking?

7. To what extent does the nature of a foreign material enveloping the grains influence the occurrence of intercrystalline cracking, for example, is iron sulfide more harmful than iron carbide?

EXAMINATION OF THE RÔLE OF OXIDATION

It will have been observed that the author is predisposed to attribute to oxidation a major part among the forces responsible for intercrystalline cracking. This charge is admitted; and Gillett's (**2**) tribute to Henry Marion Howe as a man most worthy of emulation is pleaded in justification—"His recognition of the need for theories and hypotheses to explain metallurgical phenomena, his willingness to use them as servants, as well as his questioning attitude—his refusal to accept them as masters—comprise his greatest service." On this ground it is argued that the metallurgists of an earlier day were right in accepting Rosenhain's amorphous metal theory and Jeffries' equicohesive temperature concept as working hypotheses, just as it is right that they should be rejected today. The author believes with Chipman (**19**) that the real value of a theory consists principally in that it shall get us out of more trouble than it gets us into. However, he does not ask that any theory of oxidation be accepted as an explanation of certain kinds of intercrystalline failure. He suggests, rather, that such an hypothesis would be useful in formulating an experimental program to investigate the influence of oxidation on the stress-rupture properties of metals. Manifestations of this influence are to be found all through the references to this paper.

An interesting mechanism of oxidation has been suggested by Wagner (**22**), and the essential substance of it is reported as follows by Evans (**23**):

"The parabolic law seems to be characteristic of films which contain less metal than corresponds to the stoichiometric formula. Cuprous oxide, for instance, is found to have more oxygen than corresponds to Cu_2O, especially where it is in contact with air or oxygen. It is considered to contain a certain number of cupric ions, as well as cuprous ions in the cation lattice, with a corresponding number of vacant spaces. Since a cation next a vacant space can move into it, thus leaving another vacant space into which yet another ion can move, a mechanism is provided for the transfer of copper outward through the film. But the movement of cations, if it is not to involve the electrical charging of one surface, must be accompanied by the movement of electrons. Thus the film requires both ionic and electronic conductivity, and the latter is provided by transfer of electrons between the two kinds of cations.

"Experimental evidence of the outward movement of metal has been obtained by Pfeil (**24**) in the oxidation of iron, and by Wagner (**22**) in the union of silver and sulfur."

Evans (**23**) goes on to show that the oxidation of zinc obeys a logarithmic law in keeping with simple diffusion of zinc atoms through the film. In both cases, since the cations and metal atoms are smaller than the oxygen atoms, oxidation proceeds by solution of metal ions and movement outward through the film to the surface where they react with atmospheric oxygen.

From consideration of the mechanisms outlined, an assumption that the extent and rate of oxidation is controlled by the rate of solution of ions or atoms at the film-metal interface, appears to be tenable. Further, consideration of the fact that the rate of solution of the metal ions or atoms is a function of their state of energy leads to interesting speculations. Oxidation of unstressed steel at elevated temperatures at first proceeds most rapidly at the grain boundaries (**25**), thus initiating notch effects. When the material is stressed, stress concentration would increase the energy of the atoms at these points and promote continued oxidation. Continued stress intensification by the deepening notch, and consequent increase in the energy of the atoms involved would comprise a vicious circle promoting grain-boundary oxidation and resulting, ultimately, in the intercrystalline failure actually found by experiment.

The mechanism suggested above is very similar to that proposed by Mears, Brown, and Dix (**26**) to show the effect of stress on stress-corrosion cracking. Intercrystalline failure of metals under stress at elevated temperatures and stress-corrosion cracking of metals appear to have much in common. Anderson (**27**) mentions a "threshold stress" —a minimum stress for each set of conditions, below which corrosion cracking does not occur. Wright and Habart (**28**) report that "... in all our studies of boiler tubes, superheater tubes, cracking still tubes and tubes in high-pressure chemical plant service, instances of intergranular failure of ferritic steels have been extremely rare, except in the presence of hydrogen gas." These tubes were all subjected to comparatively low stresses, while the materials of which they were made are known to be quite susceptible to intergranular failure at somewhat higher stresses. Is there, then, a "threshold stress" for the occurrence of intercrystalline failure of metals under stress at elevated temperatures? If so, it would explain the differences noted in the materials mentioned when tested under relatively high stresses and when employed in service under relatively low stresses. Also, it might explain the behavior of lead and lead alloys. If the threshold stress necessary to provide the activation energy needed to promote intergranular oxidation is so high that creep proceeds at a greater rate than oxidation, then the failures will be fully ductile, as they are in pure lead. On the other hand, if the creep rate is lowered by the presence of impurities but the activation energy required to promote intercrystalline oxidation is unchanged or lowered, then intercrystalline oxidation would proceed more rapidly than creep, and the final failure would display intercrystalline fracture. The following quotation from Carpenter and Robertson (**3**, p. 461) is pertinent in this connection:

"In general, alloying additions do not increase the resistance to intercrystalline failure to the same extent as the resistance to deformation, and in consequence the tendency of heat resisting alloys to fail in this way under conditions that they might be expected to withstand from a consideration of their behavior in tests of comparatively short duration is a problem of great practical importance."

CONCLUSION

These brief incursions into the field of speculation have been made with the object of emphasizing the need for research on the influence of oxidation on intercrystalline failure. They are not advanced with the purpose of selling a new theory to an already overtaxed audience. The author has too often observed the disruption of beautiful theories when encountered by ugly facts to place much reliance on the former until it is reasonably certain that none of the latter is lurking in the underbrush. In this case, however, there is a palpable need to seek out the facts, ugly or otherwise.

With his characteristic sagacity, Gillett (**2**) pointed out the necessity of "untangling the precipitation phenomena from those that occur in materials not subject to precipitation." The observations recorded in this discussion also indicate the necessity of untangling from the other creep phenomena the effects of oxidation; and it is respectfully submitted that this factor is entitled to priority. Moreover, it is suggested that proper evaluation of the rôle of oxidation is prerequisite to a satisfactory determination of the part played by the other factors.

Fortunately, the most difficult part of the proposed work has already been done. The apparatus devised by Jenkins and Mellor (**15**) is admirably suited to this purpose, and it only remains to apply it to the problem. The author hopes to be able to undertake this work very shortly, and he believes that by investigating pure metals first, a beginning can be made in obtaining some data relevant to the influence of precipitation hardening.

Although the question of initiating an investigation of oxidation effects in intercrystalline failure is comparatively simple, the whole problem remains complex and difficult. It will certainly not be resolved by any single individual. If the arguments presented here are sufficiently plausible to induce others to join in this work, the author will be amply repaid for the arduous hours devoted to exhuming the evidence which he has presented.

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DISCUSSION

MR. F. B. FOLEY[1] *(presented in written form)*.—Certain portions of Mr. Bleakney's paper do not hold together too well; nevertheless, he has stated the case in a rather convincing way for a more thorough study of the fundamentals which control the mechanism of failure at elevated temperatures.

The shortcomings of his Fig. 1 are not his. As it stands, it fails utterly to support the conclusions drawn from it. Experimental points in the figure, which is an accurate copy of the original, seem to indicate a definite break in the stress-time relationship in the neighborhood of 1000 hr. rupture time for the chromium-molybdenum-silicon and the S.A.E. 1015 steels, but they do not indicate, as the figure suggests and the text points out, that no such break exists for the 4 to 6 per cent chromium-molybdenum-silicon steel. Evidently no points were determined for this steel beyond about 950 hr. rupture time which is just below the point where the deviation occurred in the other two steels. This is not to deny that corrosion can cause such a break but in the interest of accuracy to point out that, as far as the 4 to 6 per cent chromium-molybdenum-silicon steel is concernd, the occurrence is not proved by the figure nor the data.

The author states that "Greenwood illustrates the progress of *intercrystalline* failure in a sample of commercial lead which broke after 470 days under a stress of 500 psi." yet later, in his summary, he asks the question: "1. Why does lead break with a *fully ductile* fracture after a test of 10,000 hr. duration?" The 470-day (11,280 hr.) *intercrystalline* failure is the only test of that order of duration cited in the paper.

Mr. Bleakney admits of a "predisposition to attribute to oxidation a major part among the forces responsible for intercrystalline cracking." It is too bad that his strong leaning in this direction had led him to pass by lightly evidence which does not support his views concerning oxidation. There is no doubt that localized grain boundary attack contributes to intercrystalline failures and that, in cases where there is pronounced chemical activity, this can be the major factor in producing such failures, but to generalize to the extent of proposing that this is the major factor underlying all intercrystalline failures is not warranted by our present-day evidence.

It is easy to agree with the author's rejection of an "equicohesive temperature" and of Rosenhain's amorphous intercrystalline cement. He discusses comments by Hanson in his 1939 Institute of Metals Lecture on intercrystalline failure as "not properly applicable to this discussion." And what has Hanson to contribute to this discussion? When aluminum was strained at 250 C.(484 F.), at the rate commonly used in mechanical testing, crystals extended progressively and a normal transcrystalline fracture eventually occurred, but when a slow rate of strain was applied, such that failure was produced at this same temperature in 220 hr., fracture was intercrystalline. Rapid straining produced by high stress caused slip lines to form within the crystals, but slow rate of strain under low stress caused deformation which was confined to the grain boundaries with no slip line formation. This same trend towards a "deformationless" intercrystalline fracture, as the time to produce failure is increased, is observed in austenitic heat-resistant alloys tested at temperatures from 1400 to 1800 F. and the phenomenon is coming to be generally observed that as the time to produce fracture increases for a given material the accompanying deformation at fracture decreases.

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[1] Superintendent of Research, The Midvale Co., Philadelphia, Pa.

I find it difficult to agree with the author's interpretation of the Sauveur high-temperature "twist" tests of iron. He finds the explanation "that gamma iron is less plastic than alpha iron at nearly the same temperature" is "easier to believe" than that "the increased plasticity (during recrystallization at the Ar_3 temperature) is contributed by recrystallization." Sauveur did more than twist specimens; he also pulled some in tension and he gave the results of these tension tests in the first Howe Memorial Lecture in 1924. His plotting showed an astonishingly low tensile strength for this steel while it was undergoing transformation. The low values are not due to the temperature of the alpha nor that of gamma, but because one phase must undergo disintegration as the other phase forms.

It is recognized by everyone that metals and alloys become less resistant to deformation as temperature increases. In the absence of any transformation, the loss of resistance can be attributed to but one thing—the increased thermal vibration or movement of the atoms. When to the normal thermal movement is added movement of atoms incident to recrystallization and grain growth, the resistance to imposed mechanical stresses is still further decreased. The resistance to deformation and the mode of failure are thus governed by the structure or "architecture" of the metal or alloy. Its strength depends primarily on the stability of the structure—not on the type of bricks of which it is built but on the ability of the bricks to stay put. In the case of steels the closer-packed face-centered cubic structure is basically stronger than the body-centered cubic form.

Where then does the chemistry of the metal or of the alloy enter into the picture? It has two functions. First of all it determines the form and the stability of the structure and, second, it determines whether the metal or alloy can exist in the environment to which it is exposed. Perhaps the second factor should be placed first, for if the metal be chemically unstable its structure is of no importance. Chemical attack can take place in two ways—as an over-all deterioration or as a localized grain-boundary infiltration. When grain boundary attack occurs it is more weakening than a general surface attack since it gives rise to stress concentration and to the phenomenon of "corrosion fatigue" or "stress corrosion."

MR. D. J. MCADAM, JR.[2]—Jeffries' general concept of an equicohesive temperature is still valid, in spite of the abandonment of the amorphous cement theory. Jeffries recognized that the "equicohesive temperature" is affected by the strain rate. However in Fig. 27 of a recent A.S.T.M. paper[3], we showed that "equicohesive boundary" is a better term than "equicohesive temperature." In that figure, abscissas represent temperatures and ordinates represent strain rates. For copper at 110 F. the equicohesive boundary is nearly parallel to the axis of abscissas. At the ordinary recrystallization temperature, however, the boundary is nearly parallel to the axis of ordinates. Since the specimens were $\frac{1}{2}$ in. in diameter, the equicohesive boundary at 110 F. was not affected by oxidation of the copper.

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[2] Chief, Thermal Metallurgy Section, Division of Metallurgy, National Bureau of Standards, Washington, D. C.

[3] D. J. McAdam, Jr., G. W. Geil, and D. H. Woodard, "Influence of Strain Rate and Temperature on the Mechanical Properties of Monel Metal and Copper," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 902 (1946).

The equicohesive boundary cannot be determined by stress-rupture tests, because the rate in such a test varies continuously. In our experiments, the rate was held approximately constant throughout the third stage, during which microscopic cracks formed and progressed to complete rupture.

MR. G. R. GOHN.[4]—The author reports that, according to the literature, pure lead fails with a normal ductile fracture, free from intercrystalline cracking when subjected to a steady stress in air. Recently we have had occasion to make creep tests on some lead of very high purity—99.995 per cent lead, the balance consisting of 0.004 per cent bismuth and 0.001 per cent sodium. Tension test specimens cut from pipe samples having a nominal thickness of 0.140 in. were subjected to initial creep stresses ranging from 2000 psi. to 100 psi. in air. So far failures have been encountered as indicated below:

Initial Stress, psi.	Time to Failure, hr.	Elongation at Failure, per cent in 3 in.	Comments
2000	<0.08	62.9	Ductile failure
1500	0.87	51.2	Ductile failure
1000	143.1	48.9	Ductile failure
800	836.	47.3	Ductile failure
600	6197	30.8	Brittle failure

The four ductile failures were predominantly transcrystalline, the brittle failure was predominantly inter-crystalline in character.

MR. H. F. MOORE.[5]—I wish to make one comment on the statement that at the University of Illinois very few transcrystalline fractures of specimens of lead alloys have been noted. Tests made since the publication of the bulletins referred to—long-time tests—have shown some very low elongations and what look like transcrystalline fractures—fractures different from the jagged intercrystalline fractures previously observed. Some of the alloy leads, notably calcium leads and antimony lead show higher strength than the relatively pure leads up to about 1000 hr. life, but still longer tests show fractures at very small elongation. Studies now in progress with other alloying ingredients show some signs of a possibility of developing a high-strength alloy which will not show so low a ductility in tests above 1000 or 2000 hr.

MR. HOWARD SCOTT.[6]—The speaker can confirm the remarks made by a previous discusser with respect to intergranular oxidation as a cause of brittle creep-rupture fractures. In the case of strong heat-resistant alloys tested at a high temperature, the ductility of some is known to increase when the time to rupture is increased by decreasing the load. If oxidation were the major cause of rupture in these alloys ductility should always decrease with load in the stress-rupture test. For this and other reasons it is highly improbable that oxidation plays an important rôle in determining the strength and ductility of high-chromium steels.

Intergranular oxidation, on the other hand, undoubtedly is the chief factor in the embrittlement of some alloys which scale rather freely. Chromium-free high-nickel and high-cobalt alloys, for example, are subject to surface crazing and cracking on hot working, a phenomenon clearly associated with preferential oxidation at grain boundaries. It is evident, then, that oxidation should be viewed with suspicion as a possible cause of brittleness at elevated temperatures, although it is not necessarily a factor in creep-rupture failures.

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[4] Bell Telephone Laboratories, Inc., New York, N. Y.

[5] Research Professor of Engineering Materials, Emeritus, University of Illinois, Urbana, Ill.

[6] Manager, Metallurgical and Ceramic Dept., Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

MR. H. H. BLEAKNEY (*author's closure by letter*).—The increasing importance being attached to the study of creep phenomena is made manifest by the growing volume of literature on the subject. The gentlemen who have favored this paper with discussion have all contributed substantially to our knowledge of it. The fact that they disagree with many of the points presented in the paper does not detract from the author's gratification that they have found it worthy of consideration.

Mr. Foley's comments have been directed more at the weaknesses of the presentation, than to any inherent fallibility in the subject matter. On this ground, the author admits to being vulnerable, but he wishes to take this opportunity to clarify those ambiguities in the paper which are the cause of the discusser's misapprehensions.

First, as regards the use of Fig. 1 in the paper, the author has not found that any objections have been raised to the postulates of White, Clarke, and Wilson with respect to the break in the curve of stress *versus* log time to rupture. Mr. Foley himself does not object and hence the author's full purpose has been accomplished, with respect to this point.

The references to Greenwood's work contain numerous examples of ductile failures of lead in tests of more than 10,000 hr. duration. Specifically, Table II on page 389 of reference 7 (*f*) reports a test on 99.9915 lead which failed after 439 days with a knife-edge fracture. It should be noted that the intercrystalline type of failure could have been brought about in this same sample by special treatment before testing. The case cited in the discussion is an example of that phenomenon. The author is chagrined to find that his use of figures, for example, 10,000 hr., has confused rather than clarified the issue. The relevant question is: Why are tests of extremely long duration required to bring about intercrystalline failure in commercially pure lead, while tests of extremely short duration are required to bring about transcrystalline failure in commercially pure copper? These characteristics cannot be reconciled, at present, with the weak grain boundary theory involved in the equicohesive temperature concept.

The author perhaps passed over Hanson's work too lightly. He did so for two reasons:

1. Hanson used 99.6 per cent aluminum and it might have been argued that this material really belonged with the group exemplified in No. 7 of the summary of the evidence contained in the paper.
2. Hanson's experiments, valuable and interesting though they were, neither contributed to nor detracted from the suggested mechanism.

The author is glad to have this opportunity to correct a definite misstatement. He would like to change the sentence "The fact that gamma iron is less plastic than alpha iron at nearly equal temperatures is an explanation of the phenomenon illustrated which is easier to believe." This should have been stated "The suggestion that gamma iron is less plastic than alpha iron at nearly equal temperatures is an explanation of the phenomenon illustrated which some may find easier to believe." In his paper on "Steel at Elevated Temperstures," Mr. Sauveur[7] says—"We infer from this that gamma iron, at the lowest temperature at which it can exist, that is, at 900 C. plus, is stronger and less ductile than alpha iron at the highest point at which it can exist, namely, 900 C. minus." The author agrees completely with Mr. Foley on this point, but, in view of the foregoing quotation, he bent over backwards, much too far, in order to avoid an argument which appeared to be controversial. He is most gratified to have Mr. Foley's confirmation on a point which he believes may prove to be of exceptional significance in the explanation of second stage creep.

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[7] Albert Sauveur, "Steel at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. 17, p. 420 (1930).

The author wishes to acknowledge his indebtedness to the writings of McAdam for much of his education on fracture of metals, limited though it be. He accepts, without hesitation, McAdam's assertion that there is an equicohesive boundary. However, there is scant and dubious evidence that this boundary is properly attributable to changing strength of grain boundary constituents. The evidence rather indicates that the equicohesive boundary is in fact the recrystallization boundary. The difference in position of this boundary may be attributed to the probability, if it is not actually an established fact, that in fine-grained metals it occurs in the direction of lower temperature, lower strain, or shorter time than in coarse-grained metals. Mr. Foley's discussion of Sauveur's experiments lends welcome support to the proposition that the equicohesive boundary is actually the recrystallization boundary.

Mr. Gohn has exposed a weakness in the author's presentation, rather than a valid objection to the subject matter. The statement that "Pure lead fails with normal ductile fracture, free from intercrystalline cracking when subjected to a steady stress in air," appears shockingly dogmatic when removed from the context. However, this statement occurred in the summary of the evidence, and the author does not suggest that evidence is proof. Actually, of course, we do not know how pure lead would fail under testing times approaching infinity. The evidence indicates that as the impurity content is decreased, the time required to bring about intercrystalline cracking is increased, and the trend suggests that with completely pure lead, intercrystalline cracking would not occur at any time. The point is, that if this is true, then intercrystalline cracking cannot be attributed to an inherent property of the metal, or its grain boundaries, but to an extraneous influence.

The figures which Mr. Gohn presents are not substantially different from those reported by Greenwood. Although 99.995 per cent appears very pure by ordinary standards, Greenwood has emphasized the marked influence on creep rates of even smaller quantities of impurities. The electrolytic lead which he employed contained only 0.0005 per cent total impurities, and Greenwood stressed the importance of melting and pouring in vacuo. Figure 1 of reference 7 (*f*), shows the creep curve of a sample of this lead, melted in air, which failed after about 76 days, with an elongation of about 23 per cent on 4 in. Oxygen absorbed during melting and pouring has a marked effect on the properties and fracture behavior of lead.

Like McAdam, Moore has unknowingly contributed greatly to the author's study of the failure of metals, and it is indeed a pleasure to have his comments. In general, they are in agreement with evidence presented in the paper. It is gratifying that the results reported by Moore agree with those of Greenwood.

Mr. Scott proposes a reason, and implies that there are others to support a statement that "it is highly improbable that oxidation plays an important rôle in determining the strength and ductility of high-chromium steels." The author agrees with this statement as far as it goes, but no further. High-chromium steels belong to a group, No. 7, in the summary of the evidence, for which the author suggested that a different mechanism than grain boundary oxidation caused brittle failure. However, Mr. Scott is also right in saying that the ductility of some heat-resistant alloys, tested at high temperatures, increases with increasing time to rupture. These alloys are very high in chromium—in some cases, nearly 30 per cent—and this high chromium is able to offset the increased susceptibility to oxidation brought about by the stress. The increased ductility is probably brought about by a metallurgical change analogous to the spheroidization of carbides in steel. Many heat resistant alloys contain only about 15 per cent of chromium, and these, in general, display marked loss of ductility as the time to fracture is increased. It is in the case of these alloys that the author suggests the need to investigate the influence of oxidation in stress-rupture tests.

The author must also agree with the statement that intergranular oxidation is not *necessarily* a factor in creep rupture failures. The whole purpose of this paper is to point out the need for investigation to find out to what extent oxidation is the controlling factor in *intercrystalline* creep-rupture failures.

The points brought out in this reply to the discussion are not in any sense intended to support or defend a special point of view. They are presented, rather, because of the need to illuminate all possible facets of the subject. The author did not intend, and has not attempted, to prove that oxidation is the cause of intercrystalline failure in creep-rupture tests. What he did intend, and what he submits that he has done, is to present a sufficient body of evidence to show that the atmosphere cannot safely be neglected as an added variable in creep-rupture tests.

PHYSICAL CHARACTERISTICS OF STEEL FOR TUBULAR PRODUCTS*

By A. B. Wilder[1]

Synopsis

It is the purpose of this paper to indicate some of the fundamental factors which influence the toughness of steel and are related to steel-making practice. Grain-size determination, high-temperature tension tests, Charpy impact tests at various temperatures of material not cold-worked, Izod impact tests of cold-worked, tapered bars, and Charpy impact tests of cold-worked tension specimens were used to evaluate the properties of the various types of steel.

Certain physical characteristics of acid bessemer, basic open-hearth, and electric-furnace steels with different deoxidation practices were investigated. Steels were selected on the basis of their commercial importance in the manufacture of welded and seamless pipe. All of the carbon steels were tested in the as-rolled condition and some of the tests were on normalized material. The electric-furnace steels were tested in the heat-treated condition normally used.

The results indicate that deoxidation practice, pipe making and shop fabricating variables, and heat treatment are the important factors which may affect the toughness of steel. The steel-making process (acid bessemer, basic open-hearth or electric-furnace) is of secondary importance.

The susceptibility of ordinary low-carbon steels to embrittlement has been the subject of many investigations. A convenient method for evaluating this characteristic, which is frequently referred to as toughness, is the Charpy or Izod impact test in which the notch impact strength of various steels at several temperatures may be compared. Another method involves a determination of the notch impact properties of cold-worked-and-aged material. The results obtained by either method depend not only on chemical composition and microstructure, but more precisely on deoxidation practice and heat treatment.

In this investigation, a comparison of the results obtained with acid bessemer, basic open-hearth, and certain grades of electric-furnace steel used in the manufacture of tubular products is reported. Grain-size determination, high-temperature tension tests and impact properties were determined. Various types of low-carbon steels are usually manufactured in the acid bessemer converter or basic open-hearth furnace, and deoxidation practices are dependent on the type of product and the experience developed in individual plants. Carbon steels, when used in the manufacture of welded pipe, are usually capped, rimmed or semi-killed. In the manufacture of seamless carbon steel pipe, killed steels are generally used and in certain instances, semi-killed steels may be successfully employed. A large ingot to provide sufficient reduction is necessary in the manufacture of semi-killed steels for seamless products to insure a sound, uniform product. Steel of electric-furnace quality for tubular products is usually an alloy grade which has been killed. Low-alloy steels made in the basic open-hearth furnace are killed steels and frequently contain nickel or molybdenum which are not oxidized during melting of the steel.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

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Steel-Making Practice:

In order to clarify our understanding of the various types of steel used for tubular products, a brief description of the steel making practice is in order. An open-hearth steel commonly used in the manufacture of welded pipe may be made by the rimmed or capped steel process. Rimmed steels are finished with a low-carbon content and not completely deoxidized so that a strong evolution of gas occurs during solidification in the molds. They have a ductile outer rim lower in carbon and phosphorus compared to the core. Bath conditions at tap for capped steels are similar to those for rimmed heats. However, sufficient deoxidizer is added to the ladle or ingot mold in capped steels so that only moderate rimming action occurs in the mold. Further, a steel cap is placed on top of the mold, and as the metal rises against the cap, it is chilled and solidifies. Capping, therefore, limits the period of gas evolution and produces a steel ingot with less rim and segregation. The ingot structure in both rimmed and capped steels consists of many deep-seated blowholes and a very small pipe cavity. The gas entrapped during solidification is in excess of that needed to counteract normal shrinkage.

Heats are sometimes finished so that the evolution of gas in the mold is closely controlled to an amount that slightly more than offsets the shrinkage caused by solidification. Steel made by this process is semi-killed and may be used in the manufacture of both welded and seamless products. Pipe cavities are minimized and the degree of uniformity is intermediate between a killed and rimmed steel. During rolling of rimmed, capped, or semi-killed ingots, the blowholes are welded.

Ingots with a dense, homogenous structure free from porosity or blowholes are produced from killed steel. In making killed steels, an excess of a strong deoxidizer such as ferro-silicon or aluminum is added to the molten steel at the end of the heat in order to reduce the oxygen content to a minimum. In steels of this type, no reaction between carbon and oxygen takes place during solidification. Careful control of the furnace charge, working the heat, and slag practice are necessary. This type of steel is commonly used for seamless pipe and has more uniform composition than any other type. Deoxidized or killed acid bessemer steel (**1**)[2] used in this investigation is not only deoxidized in the ladle with silicon and aluminum, but also with carbon which is added to the converter at the end of the blow as molten pig iron. In this special practice, a considerable portion of oxygen is removed as carbon monoxide gas by the addition of molten pig iron, producing a clean steel. Deoxidized acid bessemer steel used for the manufacture of seamless pipe is always killed and should, therefore, be distinguished from ordinary bessemer steel, which is capped or rimmed. Too frequently this distinction is not made, with the result that considerable confusion has existed in distinguishing between the two types of steel.

Due to the fact that deoxidation practices influence the susceptibility of steel to embrittlement, a considerable variation in this characteristic of steel is obtained dependent on conditions at the time of deoxidation. Also, conditions during the melting and refining periods influence the results. A further complication arises in processing which may not be related to steel-making practices but influences the embrittlement characteristics of steel. A comparison of steel in the as-rolled and normalized conditions illustrates this point.

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[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 609.

Carbon, Nitrogen, and Oxygen:

The influence of carbon, nitrogen, and oxygen has been studied by many investigators (2, 3) in an attempt to develop a better understanding of the causes for embrittlement of steel. The steel-making practice has a direct bearing on the behavior of nitrogen and oxygen (4). The amount of these elements present frequently has no relationship to the susceptibility of steels to embrittlement. The nature of occurrence has a direct bearing on the behavior of the steels. In this discussion, no attempt will be made to evaluate the significance of the individual elements. Considerable evidence has been presented in the literature to indicate that all three of the elements have, either independently or collectively, some influence on the aging and embrittlement characteristics of steel.

Low-Temperature versus High-Temperature Characteristics of Steel:

In this investigation, an attempt will be made to present data based on commercial heats of steel which meet the requirements of standard A.S.T.M. specifications for tubular products.[3] It is important to note that the embrittlement characteristics discussed in this paper which are associated with cold work are not related to specification requirements for steel pipe. The interest of the steel maker in embrittlement and aging characteristics of ordinary steels is to develop a better understanding of these phenomena. The production of steel for low-temperature services with a maximum Charpy impact value requires special deoxidation practices in conjunction with special heat treatment and frequently necessitates the use of alloy additions. Steels for this purpose are made to fine grain practice which is in contradiction to the requirements for high-temperature service. Steels for high-temperature service involving maximum resistance to creep should be made to coarse grain practice which limits the amount of aluminum added or, if necessary, alloy additions may be made to improve the creep properties. In addition to the creep properties of high-temperature steels, it is also necessary to consider the resistance of these steels to graphitization. Graphitization may be controlled by using a minimum amount of aluminum and certain alloy additions in the melting practice.

Melting steels for low-temperature or high-temperature service involves consideration of deoxidation practice, heat treatment and the use of suitable alloying elements. Nickel, for example, improves the low-temperature impact properties of ferritic steels, molybdenum the resistance to creep, and chromium the resistance to graphitization and oxidation. The production of ordinary steels with minimum embrittlement or aging characteristics after cold work and aging is a phase of steel making which is not completely understood although a number of factors related to this situation are discussed in this paper. The steel maker is in position to produce special steels suitable for low-temperature and high-temperature service, but is not in position to produce ordinary steels for these purposes due to economic factors involved. The consumer of tubular products has not developed a real need for nonaging steels and, therefore, embrittlement characteristics associated with cold deformation and aging are of secondary importance. The production of nonaging strip steels for deep-drawing stock has received considerable recognition, but this situation is not related to tubular products.

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[3] Cf. "A.S.T.M. Specifications for Steel Piping Materials," Am. Soc. Testing Mats. (1946). (Issued as separate publication.)

Our future position in regard to the embrittlement or aging of steel depends largely upon the amount of effort put forth in developing a better understanding of the problems involved. Technical and economical factors will largely control the situation. It was with these thoughts in mind that the data in this paper are presented.

MATERIALS

All of the carbon steels, with the exception of capped steels, were of a chemical analysis suitable for the manufacture of grade B seamless pipe, A.S.T.M. Specifications A 53.[4] The capped steels met the requirement for welded pipe in this specification. Capped acid bessemer and open-hearth steels are used commercially for the manufacture of welded pipe. However all of the steels discussed in this investigation were pierced into seamless pipe for comparison purposes. The embrittlement characteristics of capped steel are similar to rimmed steel and, therefore, the latter type of steel was not included in the investigation.

Deoxidized acid bessemer steel is not commercially used for the manufacture of grade A seamless pipe at the present time, although dephosphorized steel of this type would meet grade A requirements of A.S.T.M. Specifications A 53. Considerable quantities of this grade A open-hearth seamless pipe are manufactured but not included in this investigation due to the fact that a comparison with regular or deoxidized acid bessemer steel would not be significant. The embrittlement or aging characteristics of grade A low-carbon open-hearth steel should be more pronounced than the higher strength grade B open-hearth steel which contains more carbon.

Various types of grade B[4] open-hearth steel including semi-killed, silicon-killed, silicon-aluminum killed steels were studied. All of these open-hearth steels are used in the manufacture of seamless pipe, and therefore a comparison of their properties with deoxidized acid bessemer steels should be of interest. Seamless pipe is generally manufactured from either silicon-aluminum or aluminum-killed steels. The latter type of steel, with reference to embrittlement characteristics, should be intermediate between the silicon-killed and silicon-aluminum-killed steels and, therefore, was not included in the investigation.

In addition to the carbon steels, several widely used electric-furnace alloy steels (A.S.T.M. Specifications A 158[5] and A 280[6]) were included in the investigation for purposes of comparison. The embrittlement characteristics of these steels compared with deoxidized acid bessemer steel are particularly significant because the alloy steels have been successfully used for critical applications and are considered a special quality product. The carbon-$\frac{1}{2}$ per cent molybdenum was not included because $\frac{1}{2}$ per cent chromium-$\frac{1}{2}$ per cent molybdenum steel is currently used for high-temperature, high-pressure power plant installation.

The chemical analysis and austenitic grain size of the steels investigated are shown in Table I. Steels A to E were seamless pipe $5\frac{9}{16}$ in. in outside diameter

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[4] Standard Specifications for Welded and Seamless Steel Pipe (A 53 - 46), 1946 Book of A.S.T.M. Standards, Part I-A, p. 285.

[5] Tentative Specifications for Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 - 46 T), *Ibid.*, p. 918.

[6] Tentative Specifications for Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 280 - 46a T), *Ibid.*, p. 940.

TABLE I.—CHEMICAL AND PHYSICAL PROPERTIES OF STEELS.
Tension specimens Code A-E 0.505-in. Round, Code F-N 1.5-in. Strip.

Code	Grade	Treatment	Chemical Analysis, per cent							Yield Strength, psi.	Tensile strength, psi.	Elongation in 2 in., per cent	Austenitic Grain Size
			C	Mn	P	S	Si	Cr	Mo				
A.....	Capped Bessemer..	As Rolled Normalized 1950 F.	0.08	0.38	0.079	0.037	0.005	...	...	34 600 35 900	58 400 57 800	34.0 36.0	2 to 4
B.....	Capped Open-hearth..........	As Rolled Normalized 1950 F.	0.07	0.45	0.006	0.020	0.005	...		25 300 24 400	46 900 46 600	40.8 42.5	0 to 3
C.....	Deoxidized Acid Bessemer.......	As Rolled Normalized 1950 F.	0.14	0.54	0.074	0.022	0.18	...	...	39 400 42 300	68 200 64 500	32.0 34.0	7 to 8
D.....	Si-killed Open-hearth..........	As Rolled Normalized 1950 F.	0.27	0.52	0.016	0.025	0.22	...	...	35 200 ...	70 300 69 800	32.0 31.0	1 to 3
E.....	Si-Al-killed Open-hearth..........	As Rolled Normalized 1950 F.	0.24	0.86	0.013	0.021	0.23	...	...	39 200 44 900	75 100 72 500	31.0 32.3	6 to 7
F[a]...	Deooidized Acid Bessemer........	As Rolled Normalized 1650 F.	0.13 to 0.15	0.43 to 0.45	0.074 to 0.080	0.029 to 0.036	0.14 to 0.15	...	...	48 400 47 500	73 600 69 600	40.7 46.9	6 to 8
G.....	Semi-killed Open-hearth..........	As Rolled Normalized 1650 F.	0.28	0.56	0.011	0.024	0.10	...	...	39 300 43 100	72 500 71 600	39.8 41.0	1 to 2
H[b]...	Si-killed Open-hearth..........	As Rolled Normalized 1650 F.	0.25 to 0.27	0.54 to 0.57	0.015 to 0.017	0.019 to 0.022	0.20 to 0.24	...	...	45 500 42 500	76 100 75 000	38.8 39.8	2 to 3
I[b]....	Si-Al killed Open hearth...........	As Rolled Normalized 1650 F.	0.25 to 0.27	0.47 to 0.51	0.014 to 0.017	0.023 to 0.026	0.14 to 0.17	...	...	43 900 45 600	71 300 68 700	39.9 43.1	5 to 7
J.....	½% Cr-½% Mo.	Stress Relieved at 1200 F.	0.13	0.39	0.011	0.022	0.24	0.46	0.50	54 100	72 700	37.5	1 to 3
K.....	1% Cr-½% Mo.......	Stress Relieved at 1200 F.	0.13	0.47	0.016	0.019	0.17	0.98	0.53	54 900	77 200	41.8	1 to 3
L.....	5% Cr-½% Mo......	Anneal 1650 F.	0.09	0.53	0.021	0.020	0.33	4.90	0.46	30 300	68 100	48.8	5 to 7
M[c]...	Deoxidized Acid Bessemer	As rolled	0.15 to 0.17	0.39 to 0.40	0.090 to 0.091	0.026	0.19 to 0.20	...	...	49 100	74 300	38.2	
N.....	Si-Al killed Open hearth	As rolled	0.28	0.55	0.018	0.023	0.13	...	...	42 100	73 200	39.5	

[a] Six heats. [b] Three heats. [c] Two heats.

TABLE II.—TENSILE PROPERTIES OF BESSEMER AND OPEN-HEARTH STEEL AT 450 F.

Code	Grade	75 F.				Change at 450 F., per cent			
		Yield Strength, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Yield Strength	Tensile Strength	Elongation	Reduction of Area
				AS ROLLED					
A........	Capped Acid Bessemer........	34 600	58 400	34	62	−16	+16	−11	−20
B........	Capped Open-Hearth..........	25 300	46 900	41	72	−11	+33	−43	−19
C........	Deoxidized Acid Bessemer.....	39 400	68 200	32	62	−5	+32	−18	−24
D........	Si-killed Open-Hearth.........	35 200	70 300	32	57	−3	+21	−35	−25
E........	Si-Al Killed Open-Hearth	39 200	75 100	31	61	−5	+22	−35	−36
				NORMALIZED 1650 F.					
A........	Capped Acid Bessemer........	39 700	58 900	36	68	−3	+44	−11	−19
B........	Capped Open-Hearth..........	32 200	47 300	43	74	−21	+35	−37	−15
C........	Deoxidized Acid Bessemer.....	43 400	64 900	35	67	−13	0	−26	0
D........	Si-killed Open-Hearth.........	34 200	70 500	31	56	−12	+18	−34	−25
E........	Si-Al Killed Open-Hearth.....	47 500	73 900	33	64	−32	−8	−12	0

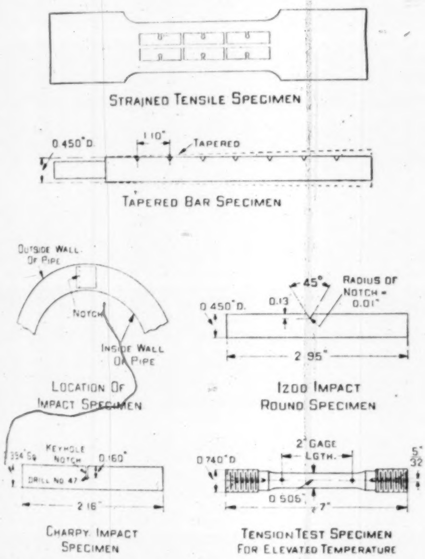
STRAINED TENSILE SPECIMEN
0.450" D.
1.10"
TAPERED
TAPERED BAR SPECIMEN
OUTSIDE WALL OF PIPE
NOTCH
INSIDE WALL OF PIPE
LOCATION OF IMPACT SPECIMEN
0.450" D.
0.13
45°
RADIUS OF NOTCH = 0.01"
2.95"
IZOD IMPACT ROUND SPECIMEN
.394" Sq.
KEYHOLE NOTCH
0.160"
DRILL NO 47
2.16"
CHARPY IMPACT SPECIMEN
0.740" D.
2" GAGE LGTH.
5/32
0.505"
7"
TENSION TEST SPECIMEN FOR ELEVATED TEMPERATURE

FIG. 1.—Schematic Drawing of Test Specimens.

with a 0.750-in. wall. Steels F to L were seamless pipe $6\frac{5}{8}$ in. in outside diameter with a 0.571-in. wall. Steel M was seamless pipe $13\frac{3}{8}$ in. in outside diameter with a 0.330-in. wall and steel N seamless pipe had a $10\frac{3}{4}$-in. outside diameter with a 0.500-in. wall. Most of the tests were made on "as-rolled" pipe except the alloy steels which were subjected to conventional heat treatment. Normalized material, although less susceptible to embrittlement, is not commercially used to any great extent and, therefore, was not considered as a basis for comparison of the various steels.

Embrittlement characteristics or toughness are frequently associated with structural grain size and therefore both coarse and fine grain materials were included in the carbon steels. The $\frac{1}{2}$ per cent chromium, $\frac{1}{2}$ per cent molybdenum, and 1 per cent chromium, $\frac{1}{2}$ per cent molybdenum steels are melted to coarse grain practice and, therefore, fine grain steels of this type were not studied.

The nitrogen content of chromium-bearing electric furnace-steels frequently exceeds that of open-hearth and acid bessemer carbon steels. Although nitrogen was not independently evaluated in this investigation, this important characteristic of electric-furnace steels should be recognized.

TEST METHODS

Tension Tests:

Tension test results in Table II are based upon standard A.S.T.M. strip and round test specimens[7] as indicated in the table and illustrated in Fig. 1. The specimens were machined from a longitudinal section of pipe. The strip specimens were $1\frac{1}{2}$ in. wide and the thickness of the pipe wall, with a 2-in. gage length. The round specimens were 0.505 in. in diameter with a 2-in. gage length. Yield strength was determined by the stress at 0.5 per cent elongation under load and, in a few instances, by the drop-of-beam method. The tension test specimen for elevated temperature in Fig. 1 was used to determine the tensile properties shown in Table II. An opening was provided on each end of the specimen for a thermocouple. The tests were conducted by the U. S. Steel Corp. Research Laboratory.

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[7] Standard Methods of Tension Testing of Metallic Materials (E 8 – 46), 1946 Book of A.S.T.M. Standards, Part I-A, p. 698.

Coarsening Temperature and Austenitic Grain Size:

The coarsening temperature was determined by exposing specimens for a period of 1 hr. at temperature, followed by air cooling. After exposure, the specimens were examined under a microscope and the actual grain size determined. Standard A.S.T.M. grain size ratings[8] were used. In addition to these tests, the austenitic grain size was also determined by the standard A.S.T.M. method[8] after carburizing for 8 hr. at 1700 F. and furnace cooling.

Impact Tests:

Impact test specimens at low temperatures of as-rolled and heat-treated material were machined from pipe samples as indicated in Fig. 1. A standard Charpy impact specimen was used for the low-temperature tests, and twelve impact tests were made on each heat and condition of treatment. Impact tests from strained tension specimens shown in Fig. 1 were two-thirds size, that is, 0.262 by 0.394 in. The depth under the notch was the same as for the standard Charpy impact specimens and three specimens were used for each degree of cold work. The longitudinal tension test specimens for straining were machined from the pipe wall. The amount of cold work was determined by percentage elongation of the tension specimen.

The round Izod impact specimen in Fig. 1 was prepared from the tapered bar specimen (5) and tested at ordinary temperatures. The tapered bar was machined from the pipe wall and drawn through a die to 0.450-in. diameter. The amount of cold work varied throughout the length of the cold-drawn bar and was determined by the percentage reduction of area of the bar.

RESULTS AND DISCUSSION

Physical Properties:

The physical properties at ordinary temperature of the steels studied in this investigation are shown in Table I. All of the steels met A.S.T.M. requirements for pipe.[3] It will be observed that the yield strength of deoxidized acid bessemer grade B steel (code F) was higher compared to grade B open-hearth steels (codes G, H, I), but the ductility as measured by elongation was similar. The high yield strength of the deoxidized acid bessemer steel was due primarily to phosphorus. The $\frac{1}{2}$ per cent chromium-$\frac{1}{2}$ per cent molybdenum (code J) and 1 per cent chromium-$\frac{1}{2}$ per cent molybdenum (code K) steels exhibited the highest yield strength of all the steels tested and exceeded the requirements of the specifications by approximately 25,000 psi. Physical properties of the carbon steels in the normalized condition were similar to the as-rolled condition.

The tensile properties of bessemer and open-hearth steels shown in Table II indicate aging characteristics in the blue-heat range. All of the steels were susceptible to age-hardening at 450 F. in the as-rolled condition. In the normalized condition, deoxidized acid bessemer and silicon-aluminum-killed open-hearth steels were not susceptible to age-hardening at 450 F. as determined by the ultimate strength and reduction of area. Yield strength and elongation in the normalized condition were lowered by the aging treatment.

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[8] Standard Classification of Austenite Grain Size in Steels (E 19 - 46), 1946 Book of A.S.T.M. Standards, Part I-A, p. 682.

TABLE III.—COARSENING TEMPERATURE OF BESSEMER, OPEN-HEARTH, AND ELECTRIC FURNACE STEELS.

Code	Grade	Initial Condition[a]	A.S.T.M. Grain Size, at								
			1550 F.	1600 F.	1650 F.	1700 F.	1750 F.	1800 F.	1850 F.	1900 F.	1950 F.
F.....	Deoxidized Acid Bessemer......	AR	7 to 8	7 to 8	7 to 8	7 to 8	7 to 8	7 to 8	7 to 8	7 to 8	5 to 8
		N	7 to 8	7 to 8	7 to 8	7 to 8	7 to 8	7 to 8	7 to 8	7 to 8	5 to 8
G.....	Semi-killed Open-Hearth.......	AR	2 to 7	2 to 7	1 to 5	1 to 4	1 to 4	1 to 3	1 to 3	1 to 3	1 to 3
		N	1 to 6	1 to 6	1 to 5	1 to 4	1 to 3	1 to 3	1 to 3	1 to 3	1 to 3
H....	Si-killed Open-Hearth..........	AR	5 to 8	4 to 8	4 to 6	3 to 6	3 to 6	3 to 5	3 to 5	3 to 5	3 to 5
		N	4 to 7	3 to 6	3 to 6	2 to 5	2 to 5	2 to 5	1 to 5	1 to 4	1 to 4
I.....	Si-Al Killed Open-Hearth......	AR	7 to 8	7 to 8	7 to 8	7 to 8	5 to 8	4 to 7	3 to 6	3 to 6	2 to 5
		N	8	8	8	8	8	5 to 8	3 to 6	3 to 6	2 to 6
J.....	½% Cr-½% Mo................	SR	7	6 to 7	5 to 7	3 to 6	3 to 6	1 to 3	1 to 3	1 to 3	1 to 3
		N	7	6 to 7	4 to 6	3 to 5	3 to 5	1 to 3	1 to 3	1 to 3	1 to 3
K....	1% Cr-½% Mo.................	SR	6 to 7	4 to 6	3 to 6	2 to 4	2 to 4	2 to 3	1 to 3	1 to 2	1 to 2
		N	7	5 to 7	3 to 5	2 to 4	2 to 4	2 to 3	1 to 2	1 to 2	1 to 2
L.....	5% Cr-½% Mo.................	ND	7 to 8	6 to 8	6 to 7	3 to 5	2 to 4	2 to 4	1 to 3	1 to 2	1 to 2
		A	7	7	6 to 7	4 to 6	4 to 5	2 to 4	1 to 2	1 to 2	1 to 2

[a] AR = as-rolled
N = normalized, 1650 F.
SR = stress-relieved, 1200 F.
ND = normalized 1650 F., stress-relieved 1200 F.
A = annealed, 1650 F.

Coarsening Temperature and Austenitic Grain Size:

Hot fabrication of pipe frequently involves temperatures exceeding the thermal critical range and therefore grain coarsening may take place in certain types of steel. A large grain size is desirable for creep resistance at certain elevated temperatures but objectionable with reference to notch impact tests at low temperature.

Grain coarsening characteristics for a number of the steels are shown in Table III. With exception of the silicon-killed open-hearth steel, normalizing did not appreciably change the grain-coarsening characteristics of the various steels. The semi-killed and silicon-killed open-hearth steels and electric furnace chromium-molybdenum steels were coarse grain at pipe fabricating temperatures. The deoxidized acid bessemer steel was fine grain at all the temperatures investigated. Silicon-aluminum-killed open-hearth steel coarsened at approximately 1850 F. and therefore grain size during fabrication would depend upon the temperature of the pipe.

Grain-coarsening characteristics are particularly significant with reference to embrittlement for all of the steels tested except deoxidized acid bessemer steel. This is a factor to be considered in the hot fabrication of pipe. The results suggest normalizing after fabrication except for the coarse grained semi-killed and silicon-killed open-hearth steels if embrittlement due to a coarse grain structure is to be avoided. Carbon steel pipe is usually marketed in the as-rolled condition. Results in Table III indicate that deoxidized acid bessemer steel had a uniform grain size over a temperature range of 1550 to 1950 F. This is significant with respect to the hot-finishing temperature of pipe as it leaves the seamless mill. Uniformity in grain size, whether the grains be large or small, indicates uniformity in the product with reference to certain fundamental properties. In this respect, deoxidized acid bessemer steel is superior to all steels tested.

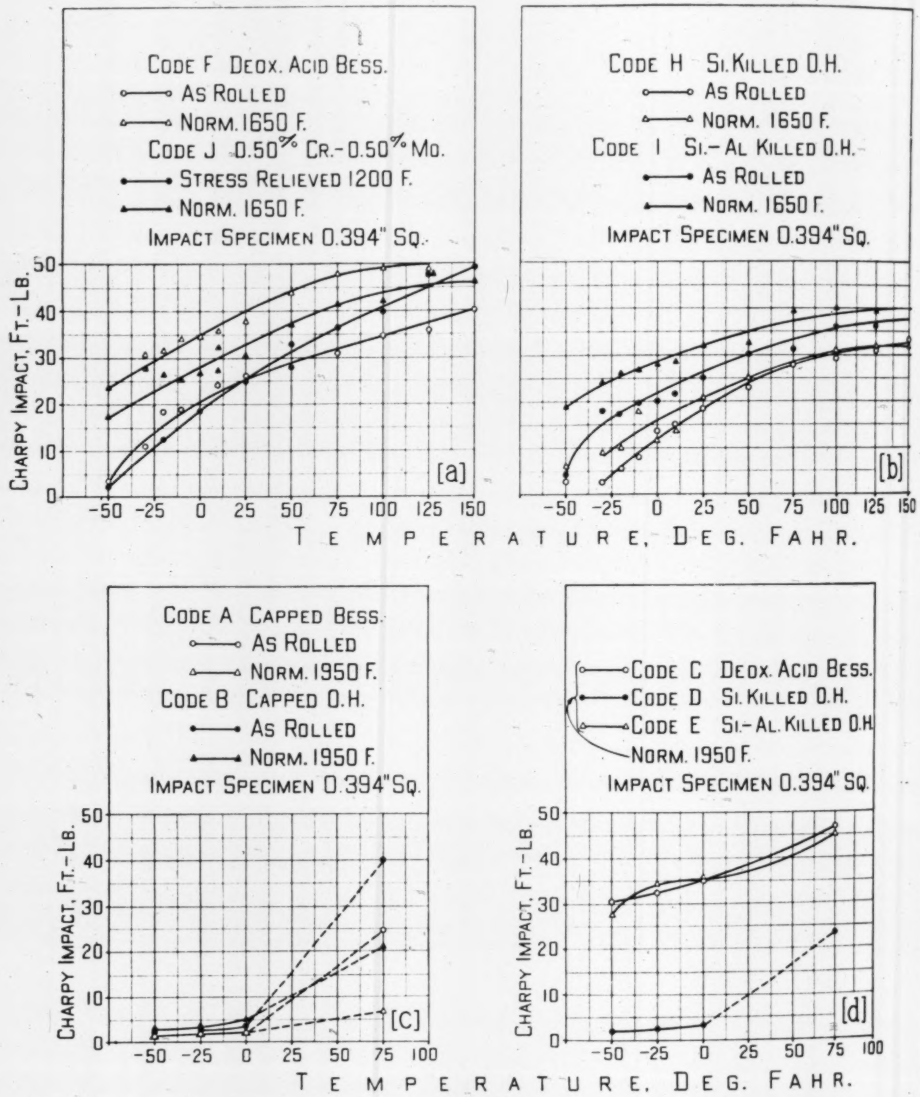

FIG. 2.—Impact Properties of Bessemer Open-Hearth and Electric-Furnace Steels.

The austenitic grain size after carburizing is shown in Table I. The capped, semi-killed and silicon-killed carbon steels were coarse grain and the silicon-aluminum-killed bessemer and open-hearth carbon steels were fine grain. The ½ per cent chromium-½ per cent molybdenum, 1 per cent chromium-

$\frac{1}{2}$ per cent molybdenum steels were coarse grain and the 5 per cent chromium-$\frac{1}{2}$ per cent molybdenum steel was fine grain. The grain size in each steel was consistent with the deoxidation practice employed.

Impact Properties of Material Not Cold Worked:

In order to evaluate the impact properties of various types of killed steel currently used for the production of large quantities of seamless pipe, a comparison of the Charpy impact properties at various temperatures is shown in Figs. 2(*a*) and (*b*). The impact specimens were machined from seamless pipe as indicated in Fig. 1. The material tested was in the condition it is normally furnished by the steel mill, and for comparison purposes pipe was also normalized. Seamless pipe, when specified for low-temperature service, is normalized and not furnished in the as-rolled condition; therefore, this treatment is of interest, particularly for service at temperatures below −20 F.

The impact properties of deoxidized acid bessemer and $\frac{1}{2}$ per cent chromium-$\frac{1}{2}$ per cent molybdenum electric furnace steels are shown in Fig. 2(*a*). The latter steel was included in the comparison because of its wide use in high-temperature, high-pressure steam power plant installations where it has given entirely satisfactory service. In the as-rolled condition, deoxidized acid bessemer steel is similar to stress-relieved $\frac{1}{2}$ per cent chromium-$\frac{1}{2}$ per cent molybdenum steel. Normalized deoxidized acid bessemer steel has superior impact properties compared to normalized electric-furnace and open-hearth steels. This may be explained partially on the basis of the uniform fine grain structure which is characteristic of the deoxidized acid bessemer steel.

The silicon-aluminum-killed open-hearth steel shown in Fig. 2(*b*) was superior to the silicon-killed open-hearth steel in a temperature range of −50 to 150 F. Normalizing both types of steel at 1650 F. improved the impact properties, but this treatment did not change the characteristics of the silicon-killed open-hearth steel to any marked degree. The impact properties of the silicon-killed open-hearth steel are, in part, related to grain size and therefore normalizing did not appreciably improve these properties. The results in Fig. 2(*b*) have long been recognized by manufacturers of seamless pipe; otherwise, it would not have been commercially possible to supply carbon steel pipe for low-temperature service.

The Charpy impact properties of various types of steel normalized at 1950 F. are shown in Figs. 2(*c*) and (*d*). A normalizing temperature of 1950 F. was selected because of the relationship of this temperature to conditions encountered in the hot fabrication of pipe. Capped steels in the as-rolled condition are also included in Fig. 2(*c*).

Although the transition temperature is not shown in Fig. 2(*c*), the results clearly indicate that as-rolled capped acid bessemer and open-hearth steels were brittle at temperatures of 0 to −50 F. Normalizing these steels at 1950 F. lowered the impact properties at 75 F., due primarily to grain coarsening and did not appreciably change the embrittlement characteristics of 0 to −50 F.

The results in Fig. 2(*d*) for material normalized at 1950 F. indicate that the silicon-killed open-hearth steel was brittle at 0 to −50 F. This is believed to be due in part to grain coarsening which was more pronounced than in the silicon-aluminum-killed open-hearth steel but did not occur at all in the deoxidized acid bessemer steel. The latter steels were not brittle at 0 to −50 F. although the impact properties were lower in this temperature range.

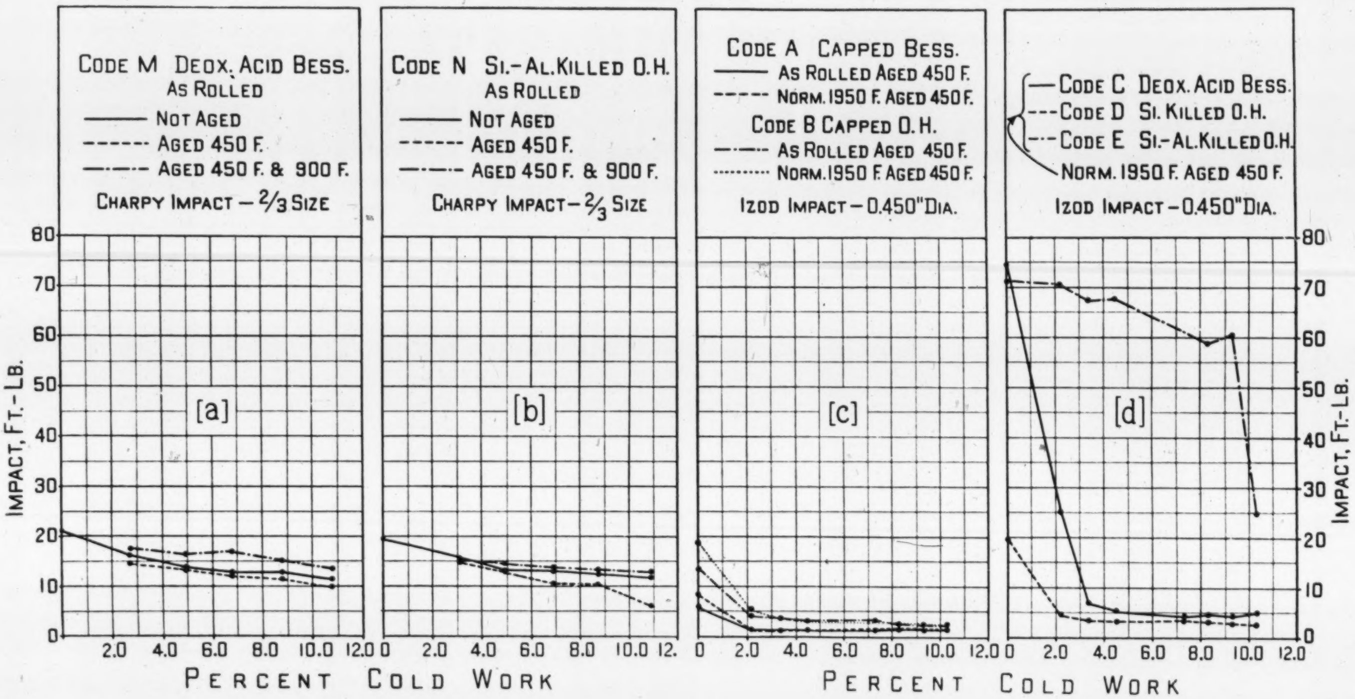

FIG. 3.—Embrittlement of Bessemer and Open-Hearth Steels.

Strain Aging of Cold-Worked Material:

The impact properties after cold working of deoxidized acid bessemer and silicon-aluminum-killed open-hearth steel pipe, Figs. 3(*a*) and (*b*), were evaluated by machining Charpy impact specimens from a strained tension specimen illustrated in Fig. 1. The material, after cold working, was tested in the following conditions: unaged, aged at 450 F., and aged at 450 F. followed by a treatment at 900 F. The purpose of the treatment at 900 F. was to illustrate the influence of overaging after cold working on the impact properties of the material. Temperatures exceeding 900 F. would probably be more beneficial and are suggested as possible means for further reducing the embrittlement effects of cold work.

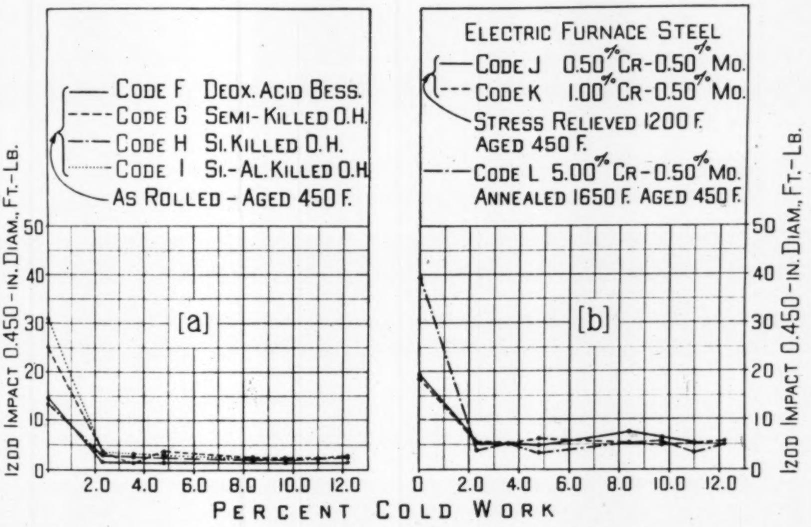

FIG. 4.—Embrittlement of Deoxidized Acid Bessemer Open-Hearth and Electric-Furnace Steels.

It will be observed in Figs. 3(*a*) and (*b*) that the impact properties for material cold worked and aged at 450 F. were at a lower level compared to unaged and overaged material. The unaged material was not strictly in the condition described due to the time required for machining the specimens. Aging characteristics of the deoxidized acid bessemer steel were similar to the silicon-aluminum-killed open-hearth steels in the as-rolled condition. It will be noted that the impact properties after cold working were essentially above 10 ft-lb.

In addition to evaluating the impact properties of pipe cold worked in tension, a number of tapered bars, Fig. 1, from other heats of steel were cold drawn through a die. The results obtained after normalizing at 1950 F. are shown in Figs. 3(*c*) and (*d*). A normalizing temperature of 1950 F. was chosen because of conditions encountered in fabrication.

The capped steels normalized at 1950 F. in Fig. 3(*c*) had low impact values after cold working and aging. The same steels in the as-rolled condition were similar to the normalized material. The silicon-aluminum-killed open-hearth steel normalized at 1950 F. in Fig. 3(*d*) was superior to deoxidized acid bessemer steel due to the high normalizing temperature. The silicon-killed open-hearth steel had lower impact properties compared with the deoxidized acid bessemer steel. Apparently, the grain-coarsening temperature was only exceeded with the silicon-killed open-hearth steel. Behavior of these steels normalized at 1950 F. is not understood.

An evaluation of the embrittlement characteristics after cold working and aging of a number of different types of steel currently used in the production of large quantities of seamless pipe is shown in Figs. 4(*a*) and (*b*). The embrittlement characteristics were determined by making impact tests at ordinary temperature on cold-drawn tapered bars machined from seamless pipe, Fig. 1. All of the material tested was in the condition furnished by the steel mill. The results, therefore, are based on properties which may be encountered in service if the material is cold worked. The various types of material included in Figs. 4(*a*) and (*b*) have given satisfactory service over a period of many years in commercial installations and, therefore, may be considered suitable for the purpose intended.

The embrittlement or strain aging characteristics in the as-rolled condition of deoxidized acid bessemer steel, semi-killed, silicon-killed, and silicon-aluminum-killed open-hearth steels are shown in Fig. 4(*a*). All of the steels have similar properties. Steel in the normalized condition, particularly if silicon-aluminum-killed, should possess higher impact properties after cold working compared with as-rolled material. However, normalized carbon steel pipe is not used for ordinary piping installation and therefore was not included in this part of the investigation. It should be noted that the impact results of the tapered bar specimens were considerably lower than those obtained from the cold-worked tension specimens.

Impact properties after cold working as-rolled material may vary appreciably if a large number of heats involving different deoxidation practices are investigated. Variations in finishing temperature on the seamless mill and other factors encountered in the manufacture of pipe from a steel ingot may influence embrittlement characteristics of the material. It is for these reasons that results reported in the literature have been largely based on normalized material as the influence of variations in the rolling of steel on embrittlement are little understood. When the steel pipe is normalized, certain properties imparted to the material may originate in the normalizing treatment. The factors involved in this treatment have never been thoroughly evaluated with reference to embrittlement due to cold deformation. Normalizing, however, has served a useful purpose in providing a means for evaluating the influence of certain steel making practices on embrittlement after cold working which otherwise would not have been evaluated in the as-rolled condition. This approach, however, should not be exclusively used in the evaluation of the susceptibility to embrittlement or aging after cold deformation of material which is used in the as-rolled condition. In this latter case, we are more concerned with the actual properties of the material rather than the relationship of these properties to the steel making practice.

The embrittlement or strain aging characteristics of cold-worked $\frac{1}{2}$ per cent chromium-$\frac{1}{2}$ per cent molybdenum, 1 per cent chromium-$\frac{1}{2}$ per cent molybdenum, and 5 per cent chromium-$\frac{1}{2}$ per cent molybdenum electric furnace steels are shown in Fig. 4(*b*). The steels were selected because large quantities of seamless pipe rolled from these alloys are used for elevated temperature service. This material was tested in the condition furnished by the steel mill. It will be observed in Fig. 4(*b*) that the electric furnace steels were brittle after cold working, but the impact strength was at a higher level compared to the carbon steels in Fig. 4(*a*). The chemical composition of these steels is probably responsible for the higher level of impact properties.

SUMMARY AND CONCLUSIONS

The embrittlement or strain-aging characteristics of acid bessemer, open-hearth, and electric-furnace steels with various deoxidation practices have been discussed. All of the material investigated was seamless pipe and most of the results were reported for material in the as-rolled condition. The conclusions, based on the steels investigated, are as follows:

1. Chemical and physical properties met A.S.T.M. specification requirements for steel pipe.
2. The carburized austenitic grain size at 1700 F. was consistent with the deoxidation practice employed, but independent of the steel making process. Capped, semi-killed, and silicon-killed steels were coarse grain and the silicon-aluminum-killed steels were fine grain.
3. Deoxidized acid bessemer steel had the highest grain-coarsening temperature and the most uniform grain size.
4. Tension test results at 450 F. indicated that as-rolled material was susceptible to age hardening. After normalizing at 1650 F., the deoxidized acid bessemer and silicon-aluminum-killed open-hearth steels did not age harden at 450 F.
5. The Charpy impact properties of deoxidized acid bessemer and silicon-aluminum-killed open-hearth steels normalized at 1950 F. were superior to the capped and silicon-killed carbon steels.
6. Charpy impact properties in the as-rolled condition and in the absence of cold deformation were similar in a temperature range of −50 to 150 F. for the deoxidized acid bessemer, silicon-aluminum-killed open-hearth and electric furnace steels. In the normalized condition (1650 F.) deoxidized acid bessemer steel had the highest impact strength.
7. Strain-aging characteristics after cold working pipe in the as-rolled condition were similar in the deoxidized acid bessemer, semi-killed open-hearth, silicon-killed open-hearth and silicon-aluminum-killed open-hearth steels. The capped acid bessemer and open-hearth steels were at a slightly lower level and the electric furnace steels were at a slightly higher level compared with the other steels.

Acknowledgment:

The author acknowledges the encouragement received from E. N. Sanders, Vice President in Charge of Operations, and the assistance and cooperation of the operating personnel of the National Tube Co. in conducting this investigation. He is particularly indebted to W. B. Kennedy, Chief Metallurgist of National Works, under whose direction most of the experimental work was conducted.

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DISCUSSION

Mr. Perry R. Cassidy.[1]—It is high time that engineers consider the required characteristics of steels for a given application and stop relying upon nominal and arbitrary factors hallowed by long use. This paper is an excellent example of what can be done to determine the effect of important variable factors in steel melting and heat treatment upon the characteristics of pipe and tubes. The fabrication and service requirements can be used to select the most favorable factors to be specified. The paper shows that chemical analysis alone is not the most important factor.

The word "bessemer" has become through the years an indication of undesirable qualities for many steel applications. Such fixed habit will require time and continued effort to overcome. The author and his associates have assumed this difficult task intelligently, industriously, and effectively. In E. C. Wright's paper before the Am. Inst. Mining and Metallurgical Engineers in 1944[2] and in the present one, complete data are given from which engineers may judge for themselves the relative merits and suitability of steels made by definite processes.

Engineers responsible for safety codes, as distinguished from those whose function it is to purchase and inspect, are looking towards a change in philosophy to simplify and rationalize their rules for approval of materials. Such engineers seek means of defining limits within which materials may be used for given purposes without confining chemical analysis or other nominal factors too narrowly.

Present specifications consider the meeting of minds of purchaser and producer, and therefore must be definite and limited in range and scope.

Those responsible for formulating safety codes should deal only with the factors of materials which affect safety, and leave great flexibility and latitude to the fabricator and the material producer in mutual selection and agreement upon definite specifications. Data such as those presented in this paper will be of great help in selecting the factors that affect safety and in deciding upon the limits which should apply.

Mr. A. B. Wilder (*author*).—The author appreciates the encouragement given to the type of work described in the paper by the remarks of Mr. Cassidy. An effort should be made to evaluate factors beyond the scope of standard specifications so that engineers will have an opportunity to define more clearly conditions for proper use of the material.

In this investigation emphasis was placed on the influence of deoxidation practice on impact properties of certain types of steel. A similar approach might be applied to the creep strength of steel. Deoxidation of steel is not an exact science; however, sufficient information is available to permit a more intelligent use of materials if an effort is made to apply our knowledge to the development of essential data. Success along these lines depends not only upon experience and technological information but, also, as Mr. Cassidy points out, upon a sound philosophy which takes into consideration the future as well as the present.

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[1] Executive Assistant, The Babcock & Wilcox Co., Boston, Mass.

[2] E. C. Wright, "Manufacture and Properties of Killed Bessemer Steel," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 158, p. 107 (1944).

MR. A. HURTGEN.[3]—The tensile properties as described in Mr. Wilder's paper determined on steels suitable for welded and seamless pipe of A.S.T.M. Specifications A 53 and A 106[3a] in the range from room temperature to 500 F. give us a picture of what happens to the ductility under the combined effect of temperature and stress. I shall refer to this test as the blue-brittle peak-tension test. To appraise the steels on the basis of the results, it is desirable to do so in two steps.

First, consider the loss (minus) or gain (plus) in the ductility values for each test within the temperature range mentioned. If these are all positive or all negative values the response of all steels tested may be said to be similar. For the second step, we must have some standards to use as a yardstick defining minimum acceptance limits for ductility, for instance, 20 per cent elongation, 40 per cent reduction of area, etc.

In this connection I wish to refer to the tests conducted by the subcommittee of the Ferrous Materials Committee, A.S.M.E. Boiler Code under the chairmanship of the writer. These included tests on capped open-hearth, capped bessemer, deoxidized acid bessemer, and killed open-hearth steels manufactured to specifications for welded and seamless pipe (A 53 and A 106). The peak-tension on all the steels occurred within a 100-deg. range or between 400 and 500 F. The response for as-rolled steels was similar for all steels in that the tensile strength passed through a peak; both the percentage elongation and reduction of area passed through a valley, suffering a ductility loss as compared to the results at room temperature.

On the basis of the peak-tension we can say all the steels are similar. Whether the actual minimum values obtained for elongation and reduction of area are sufficient, calls for the yardstick of acceptable limits. If such limits have not been provided beforehand the results of the test have little significance. The committee tests showed an increase in tensile strength over room-temperature results of 13 to 41 per cent; a loss in elongation of 4 to 43 per cent; and a loss in reduction of area of 14 to 29 per cent.

On the basis of loss in percentage elongation the steels are classified in order of merit as follows:

	Loss, per cent	Lowest Actual Figure, per cent
1. Capped Acid Bessemer	4	26
2. Killed Open-Hearth	28	22
3. Deoxidized Acid Bessemer	31	21
4. Capped Open-Hearth	43	23

On the basis of loss in percentage reduction of area the steels are classified in order of merit as follows:

	Loss, per cent	Lowest Actual Figure, per cent
1. Capped Acid Bessemer	14	52
2. Capped Open-Hearth	15	55.5
3. Killed Open-Hearth	17	48.6
4. Deoxidized Acid Bessemer	29	43

In these tests the lowest elongation and reduction of area does not always occur at the temperature of peak-tension.

The deoxidized acid bessemer and killed open-hearth were fine grain (No. 6 to No. 8 hypereutectoid[4]) and the capped bessemer and capped open-hearth were coarse grain (No. 2 to No. 5 hypereutechtoid) on McQuaid-Ehn. In this connection it was significant to note that the capped steels showed a loss of yield strength (0.2 per cent offset) from 11 to 13 per cent, whereas the deoxidized or killed steels showed an increase of yield strength (0.2 per cent offset) from 10 to 25 per cent in the blue-brittle tension tests independent of the process, whether bessemer or open-hearth.

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[3] Chief Metallurgist, Henry Vogt Machine Co., Louisville, Ky.

[3a] Tentative Specifications for Seamless Carbon-Steel Pipe for High-Temperature Service, 1946 Book of A.S.T.M. Standards, Part I-A, p. 907.

[4] Standard Classification of Austenite Grain Size in Steels (E 19 - 46), *Ibid.*, p. 682.

The foregoing comments apply to the "as-rolled" condition for all the steels mentioned.

Izod type notched-bar work-brittleness tests described by Graham and Work[5] also were used to determine whether the steels possessed any characteristics attributable to the steel making process and deoxidation practice. The bars were cold drawn through a carboloy die at a rate of 1 in. per min., notched, aged 1 hr. at 450 F. and tested. The specimen was rotated 30 deg. between successive notches to detect any possible difference due to orientation of stress. As in the blue-brittle tension test, it is desirable to appraise the steels on the basis of these results in two steps.

First, consider the loss (minus) or gain (plus) in impact values for each steel mentioned. If these are all minus values representing a loss of impact, the response of all the steels tested may be said to be similar.

For the second step, we again must have some standard to use as a yardstick defining minimum acceptance limits for Izod work-brittleness values. These we do not have and furthermore we have practically no precedent upon which to decide such as we have in referring to elongation and reduction of area. We have been unable to reach a satisfactory agreement even for low-temperature equipment based on impact values. The results of these tests indicated that all steels tested are similar in that the impact values suffered a loss as a result of cold work and aging, excepting that the killed open-hearth steel goes through a maximum at 0.7 per cent cold work.

The second step involved a classification as to the actual minimum impact values for which no yardstick for evaluation is available.

On the basis of loss in impact values as determined at room temperature on "as-rolled" steel by the work-brittleness test, the steels may be classified as shown in order of merit:

1. Killed open-hearth, minimum impact value at about 6.3 per cent cold work, suffered a loss of 66 per cent.
2. Capped open-hearth, minimum impact value at about 4.5 per cent cold work, suffered a loss of 86 per cent.
3. Deoxidized bessemer, minimum impact value at about 4.5 per cent cold work, suffered a loss of 94 per cent.
4. Capped bessemer, minimum impact at about 2.6 per cent cold work, suffered a loss of 96 per cent.

The foregoing applies to the as-rolled condition for all the steels tested.

Finishing temperatures on all steels were carefully controlled and recorded and in all probability are much more uniform than would be encountered in the usual practice of rolling pipe skelp or working seamless pipe. No determinations were made for grain-coarsening temperatures; however, it is probable that this would be influenced to a variable extent by variations in finishing temperatures concurrent with variations in the deoxidation practice and manufacturing process.

The evidence indicates that the state of oxidation of the steel and the subsequent deoxidation practice are of at least equal, if not more, importance than the steel manufacturing process, other things being nearly equal.

MR. WILDER.—One of the most extensive investigations of the strain-aging characteristics of acid bessemer and open-hearth steels has been conducted by the Hurtgen Committee of the A.S.M.E. Boiler Code Subcommittee on Ferrous Materials. The results of this investigation which includes capped and killed steels have never been published and the author appreciates the reference to this work by Mr. Hurtgen.

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[5] H. W. Graham and H. K. Work, "A Work-Brittleness Test for Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 571 (1939).

MR. G. H. ENZIAN.[6]—Mr. Wilder has presented interesting data supporting his contention that deoxidation practice, heat treatment, etc., are the important considerations influencing the toughness of steel, with the steel-making process of secondary importance. It is of direct practical significance that the deoxidized acid bessemer and silicon-aluminum killed basic open-hearth steels discussed in the paper have similar properties when tested after similar treatments.

Another way of viewing the question of differences in the properties of various steels is that the steel-making process generally, but not necessarily, imparts certain characteristics to the steel, which are associated with the chemical composition. As the result of a considerable amount of work based on this generalized premise, we believe that the basic reasons for differences in the properties and behavior of steels lie in the precise details of the composition. By this is meant not only the chemical composition of the material in terms of all the elements present, but also the manner in which those elements may be in chemical combination or in solution. For example, it has been shown (reference 4 of the paper) that nitrogen, when present essentially in the form of aluminum nitride, does not promote strain-sensitivity. Thus the amount of nitrogen in the steel (or the steel-making practice by which it was introduced) is of secondary importance compared to the nature of its occurrence.

MR. WILDER.—The author is essentially in agreement with the remarks of Mr. Enzian. Normally the chemical composition of steel is expressed in terms of the usual chemical elements such as carbon, manganese, phosphorus, sulfur, and silicon. The precise details of chemical composition, particularly with reference to the mode of occurrence of elements such as nitrogen or oxygen is significant because the total nitrogen or oxygen present in steel with reference to embrittlement is of secondary importance. In order to determine the nature of chemical compounds and solid solutions of oxygen, nitrogen and other elements in steel, complex analytical procedures are involved which require further development before the required accuracy is recognized. The control of these variables in the steel making practice further complicates the problem. From the viewpoint of steel making, the National Tube Co. is conducting extensive experiments that will increase our knowledge of this particular subject.

MR. J. HEUSCHKEL[7] (*by letter*).—The paper is one of a type rather badly needed by industrial users of steel who do not themselves have direct contact with current manufacturing methods and practices and who, at the same time, assume a real responsibility for the quality or service performance of the final welded, or otherwise fabricated, product. More such information is needed for various sheet, plate, and bar products.

Paralleling the author's statement, I have often observed in welded joint test work that deoxidation practice has a pronounced influence upon the final properties, particularly under notched bending conditions. Unfortunately in too many cases the ordinary user of steel does not know anything about the practices employed for the production of any particular lot of steel. Such information is seldom furnished by the manufacturer.

I believe the statement to the effect that nickel improves the low-temperature impact properties of ferritic steels should be qualified somewhat. After welding and then testing at low temperatures under concentrated stress conditions such steels may be "tough" or brittle, depending upon several conditions. The mere addition of nickel does not, by itself, assure good low-temperature properties on welded joints.

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[6] Research Engineer, Jones & Laughlin Steel Corp., Pittsburgh, Pa.

[7] Welding Section, Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

I concur in the statement that the presence of nitrogen in bessemer steels is a most important factor. It has been my experience in weld-testing bessemer steels that those containing the highest nitrogen content give the poorest performance measured on final deformation.

With reference to the results obtained from the impact tests on varying degrees of cold work, it would be of interest to know whether the static load and deflection values were also lowered by the cold work where the same type of specimen was tested under bending conditions.

MR. WILDER.—The author appreciates the remarks of Mr. Heuschkel. With reference to the use of nickel for improving the impact properties of ferritic steels, practice in the pipe industry is to normalize the material. This treatment may be followed by stress relieving.

In our experience with welding deoxidized acid bessemer steel, we have found no relationship between the nitrogen content and mechanical properties. This is probably due to the use of aluminum for deoxidation. In ordinary acid bessemer steels, a very limited amount of aluminum is used for deoxidation due to the fact that steels of this type are usually capped or rimmed. We have no data regarding the static load and deflection under bending conditions.

CREEP AND CREEP-RUPTURE TESTING*

By G. V. Smith,[1] W. G. Benz,[2] and R. F. Miller[3]

The more rigorous requirements with respect to metal for use at high temperatures have emphasized the need for reliable information to serve as a basis for selection of the most suitable metal for each use; consequently a more widespread interest in all details of the appropriate methods of measurement of high-temperature properties has recently been developing. In this paper are described the apparatus and experimental procedure now in use at this laboratory.

If the results of creep or creep-rupture tests are to be significant, careful attention must be paid to all experimental details of preparation of the specimens, uniformity and control of temperature, and measurement of the several properties involved in the contemplated use of the metal. The apparatus and methods which we have developed to meet these conditions successfully are, therefore, described in considerable detail which would be required by anyone who contemplates a test program of this character.

The creep stand is patterned after similar apparatus at the Crane Co., Chicago, Ill., and at Battelle Memorial Institute, though certain modifications have been made. The mode of measuring extension, however, as well as certain testing techniques, are unique, and, therefore, are described in more detail.

No effort has been made to review the literature or to compile a bibliography on this subject, since to do so would unduly lengthen the paper by including data which have been published elsewhere.[4]

The Nature of Creep

There are today many applications of metals which involve long-time service at elevated temperature—in power-generating stations, in chemical processing, in oil-cracking stills, and of recent interest, in gas turbines. Unfortunately, the ordinary tension test, so widely used for evaluating the load-carrying ability of metals at ordinary temperature, cannot be similarly evaluated for elevated temperature service even though the test be made at the temperature of interest. The reason for this lies in a fundamental difference in behavior of metals at normal and at elevated temperatures. At ordinary temperature, the extension which attends the stressing of a metal ceases, for practical purposes, immediately after application of the load, provided that the load is not sufficiently great to produce failure,

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Research Laboratory, United States Steel Corp., Kearny, N. J.

[2] Formerly with Research Laboratory, United States Steel Corp.; now with Air Reduction Co., Murray Hill, N. J.

[3] Formerly with Research Laboratory, United States Steel Corp.; now with Carnegie-Illinois Steel Corp., Pittsburgh, Pa.

[4] For example:

(1) J. J. Kanter, "The Creep of Metals and Alloys," *Metals Handbook*, Am. Soc. Metals, Cleveland, Ohio.

(2) D. Hanson, "The Creep of Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 133, pp. 15–57 (1939).

(3) H. W. Gillett, "Some Things We Don't Know About the Creep of Metals," *Ibid.*, Vol. 135, pp. 15–58 (1939).

(4) J. A. Fellows, E. Cook, and H. S. Avery, "Precision in Creep Testing," *Ibid.*, Vol. 150, pp. 358–372 (1942).

nor the melting point so low, such as with lead, in relation to the common structural metals, that ordinary temperature is in reality an "elevated" temperature. On the other hand, at elevated temperature, extension continues even under low stress. This slow extension under stress is termed creep, and the rate at which it occurs in a given material is dependent upon stress, temperature and the history of the material, increasing always—and usually more than proportionately—with stress and with temperature.

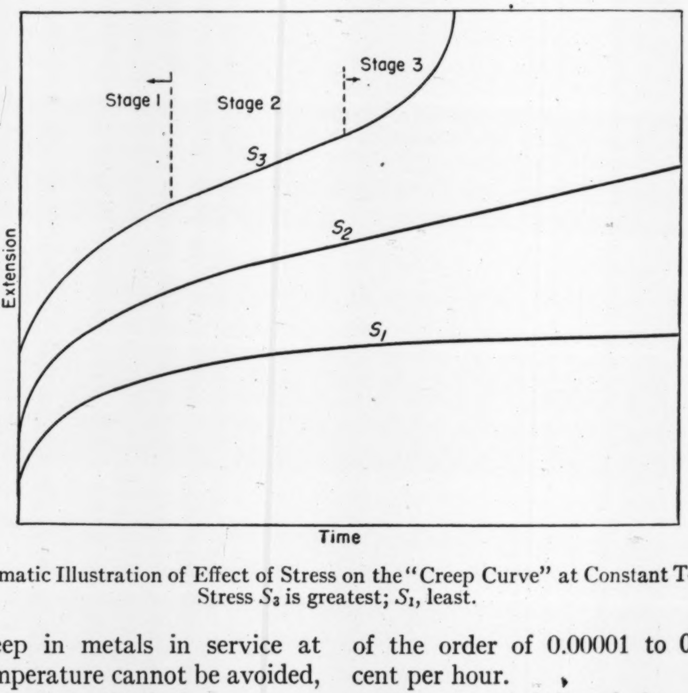

FIG. 1.—Schematic Illustration of Effect of Stress on the "Creep Curve" at Constant Temperature. Stress S_3 is greatest; S_1, least.

Since creep in metals in service at elevated temperature cannot be avoided, the engineer must take it into account in designing equipment; consequently, he needs quantitative information on the rate of extension produced in the metal at the contemplated operating temperature by the stresses involved in his design, and on the probable length of service under these conditions before rupture occurs. To provide information on these points, creep and creep-rupture tests are made. As explained more fully in the following sections, the creep and creep-rupture tests differ only in that the latter is conducted at a stress sufficiently great to produce rupture during the test.

The Creep Test:

In principle the creep test is simple—a specimen is heated to the temperature of interest, a constant load is applied (generally by dead weights and a lever), and the resulting slow extension is measured periodically. In practice, however, it requires great experimental care because the extension is very small, of the order of 0.00001 to 0.0001 per cent per hour.

The general form of the curve representing the elongation of a metal under constant stress at constant elevated temperature is illustrated by the schematic curves in Fig. 1, for the stresses S_1, S_2, S_3 respectively, S_3 being greatest and S_1 least.[5] When the stress is high (curve S_3) there are three fairly distinct stages of elongation: (1) an initial period of rapid but diminishing rate; (2) a period of substantially constant rate; (3) a period of increasing rate which is a portent of eventual failure. Conditions such that the actual curve is like curve S_3 within a period of 1000 hr. or less are chosen for creep-rupture tests; whereas in creep testing, the aim is to select the stress so that the curve, over a period of, say 3000 hr., will resemble curve S_2 or even curve S_1. The rate of extension during the second stage is utilized in estimating creep-strength, defined usually as the stress which produces a creep-rate of 0.1 per cent per 1000 (or occasionally, per 10,000) hr. Such a small rate is assumed to persist during the service life of the metal; when the rate continuously diminishes in course of the test, as sometimes happens, creep strength is commonly based upon the observed rate over the last 500 hr. Consequently in the creep test, the third stage of the typical curve does not generally occur (except possibly in preliminary experiments), because the stress chosen is purposely made small enough to prevent it; but the possibility of its occurrence at some subsequent time must be borne in mind.

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[5] It should be recognized that the stress does not really remain constant throughout the test, since while the load remains constant, the cross-sectional area of the specimen continually decreases. In the creep test, where the elongation usually does not exceed 1 or 2 per cent, this is of no consequence.

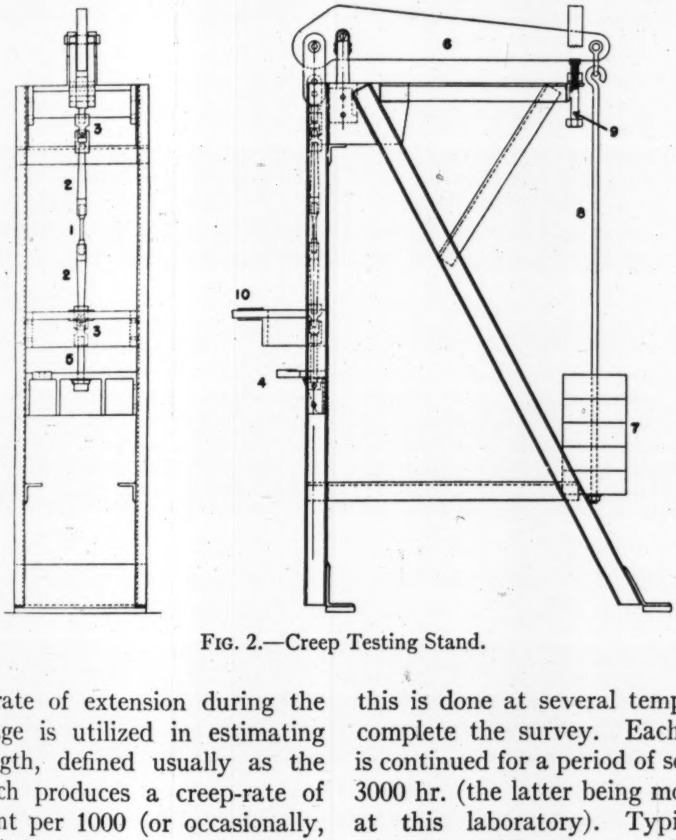

FIG. 2.—Creep Testing Stand.

In practice, at each temperature level each material is tested at several stresses, so that interpolation may be made to obtain the stress for a specified creep rate at that temperature; and this is done at several temperatures to complete the survey. Each creep test is continued for a period of some 1000 to 3000 hr. (the latter being most common at this laboratory). Typical results are evaluated in a later section.

The Creep-Rupture Test:

In the creep-rupture test, frequently called the stress-rupture test,[6] the third stage of the creep curve of Fig. 1 does occur, for, as its name indicates, this test is continued to failure; in fact, the only difference between the creep test and the creep-rupture test is that the latter is conducted at sufficiently high stress so that fracture results during test. A series of specimens are tested each at a different stress so as to rupture at different elapsed times, varying generally up to 1000 hr. or more; these data are interpolated or extrapolated to a time corresponding to the service life. In this test, the time for rupture, rather than rate of extension or even total extension, has been of primary interest, but investigators are, more and more, realizing the value of the latter quantities, particularly extension-rate.

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[6] The term stress rupture is unnecessarily confusing in that it indicates a greater difference from the creep test than actually exists. The term should be, and is, being discarded.

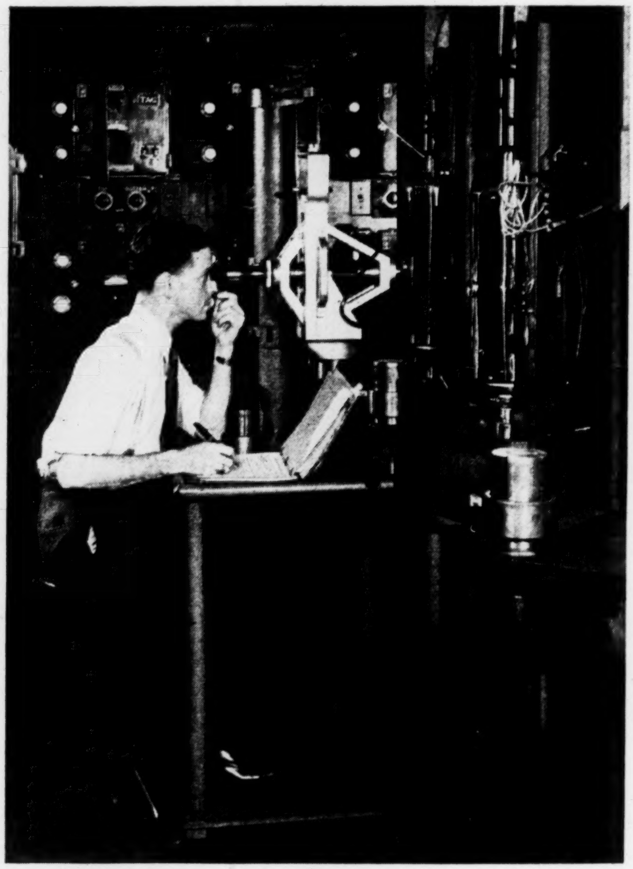
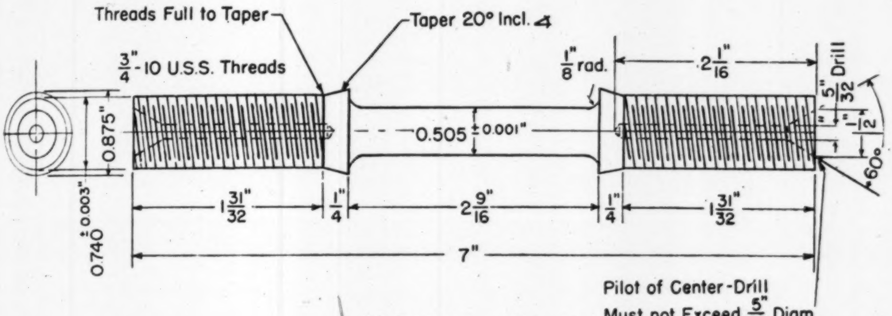


FIG. 3.—Technician Measuring Extension of Creep Specimen.

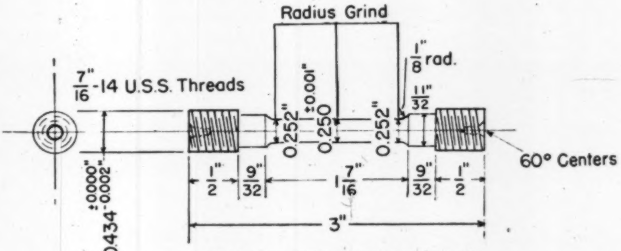
Application of the Measurements:

Whether data on creep (rate of extension) or on creep-rupture (time for failure) are more to be desired in a particular instance depends on the contemplated application of the metal. Generally, when the service life is not long and distortion of the structure is of little concern, creep-rupture data are of primary and sufficient interest.

When, on the other hand, as in the steam turbine, only very slight deformation can be tolerated, knowledge of creep rate is necessary. However, creep-rupture data are also needed in such applications because, though creep rate permits the engineer to design so that only tolerable distortion occurs, it is possible that rupture will occur within this limitation.


Threads Full to Taper
Taper 20° Incl. ∢
3/4"-10 U.S.S. Threads
1/8" rad.
2 1/16"
5/32" Drill
1/2"
1
60°
0.875"
0.505 ± 0.001"
0.740 ± 0.003"
1 31/32"
1/4"
2 9/16"
1/4"
1 31/32"
7"
Pilot of Center-Drill Must not Exceed 5/32" Diam.

(a) Creep-test specimen 1


Radius Grind
1/8" rad.
7/16"-14 U.S.S. Threads
11/32"
0.252"
0.250 ±0.001"
0.252"
60° Centers
0.434 ±0.000" -0.002"
1/2"
9/32"
1 7/16"
9/32"
1/2"
3"

(b) Creep-rupture test specimen 1

FIG. 4.—Dimensions of Test Specimens.

METHODS AND APPARATUS

This laboratory has been conducting creep tests for over ten years and has recently installed equipment for creep-rupture tests; currently, there are facilities for testing 12 creep and 8 creep-rupture specimens simultaneously. In the sections which follow, a description is given of how the tests are conducted with the degree of accuracy necessary if the results are to be significant, with all pertinent details concerning the various aspects of: preparation of the specimen; method of loading; temperature control; measurement of extension.

CREEP TESTING

The Test Stand:

The stand in which the creep test is conducted is shown in Fig. 2. It consists of a steel framework approximately 20 by 45 by 80 in. surmounted by a simple lever system, through which the specimen is loaded by means of dead weights, a specimen anchorage, and a mounting for the microscope for measuring the extension. Figure 3 shows this microscope and the heating furnace in place; both these items are described in subsequent sections.

Referring to Fig. 2, the specimen *1* mounted in the pull rods *2* is threaded into a pair of universal joints *3* which serve to minimize, but unfortunately not to entirely eliminate, nonaxial loading. The lower universal joint is attached to the anchorage bracket *4* by a spherically seated bolt *5*; the upper is connected to the short arm of the lever *6*. This lever has an arm ratio of 9 to 1 and a sensitivity of several grams so that the possible error in stress is negligible. The loading weights *7* are carried on a weight hanger *8*; prior to loading the specimen, the lever is supported in position by a thrust bearing *9*. To support the furnace around the test specimen, a special collar (not shown) is screwed onto the lower pull rod. The microscope for measuring extension of the loaded specimen is mounted, as shown in Figs. 3 and 13, on a special post attached to yoke *10*. The pull rods are machined from castings of the following composition: carbon, 1.5 per cent maximum; silicon, 1 per cent; chromium, 30 per cent; nickel, 30 per cent; tungsten, 4 per cent; they have proved entirely satisfactory.

The Creep Test Specimen and Its Preparation:

Prior to machining the specimen, a blank of bar stock, at least 1 in. in diameter or thickness and 8 in. long, is heat treated according to desired practice, and a $\frac{1}{2}$-in. coupon is then cut from each end to serve for metallographic examination of the initial structure. The remaining 7 in. is machined accurately to the specifications shown in Fig. 4(*a*). The end holes are for thermocouples, and the pair of tapered shoulder sections to carry the units on which the extension is measured, as described below.

To measure the extension of the specimen during test, a microscope (described below) is sighted upon reference marks engraved on polished platinum beads attached on extensometer units which are secured to the shoulders of the specimen. The platinum beads are brazed or welded on the arms of the unit *B*, Fig. 5, which when thus mounted, refer the extension between the shoulders to a central point for observation (see also Figs. 6 to 8). From periodic observations of a reference mark on each of the platinum beads the extension-time curve is constructed.

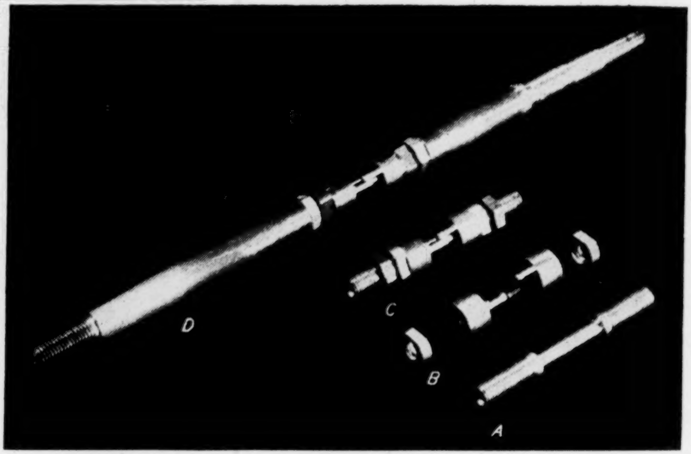


FIG. 5.—Mounting of the Extensometer Units.

A—Test specimen
B—Extensometer units
C—Units mounted on specimen
D—Final assembly in pull rods

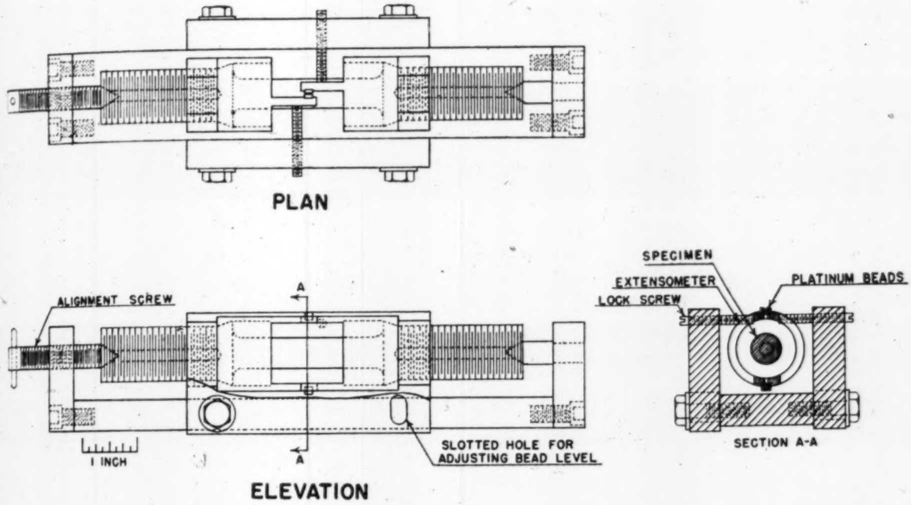

PLAN
SPECIMEN
EXTENSOMETER
PLATINUM BEADS
LOCK SCREW
ALIGNMENT SCREW
A
A
1 INCH
SLOTTED HOLE FOR ADJUSTING BEAD LEVEL
SECTION A-A
ELEVATION

Fig. 6.—Jig for Polishing Extensometer Units After Mounting on Creep-Test Specimen.

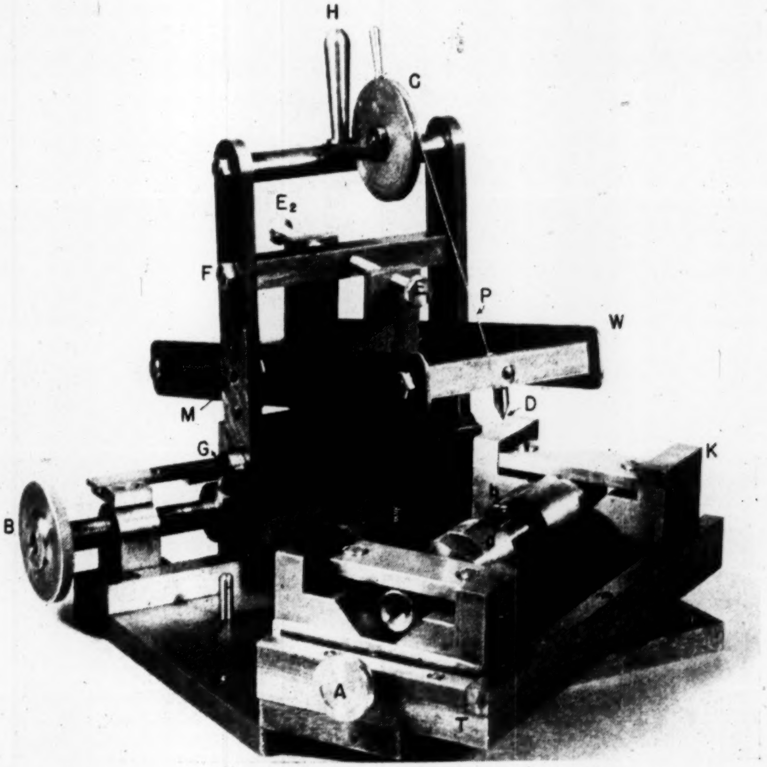

H
C
E_2
F
P
W
M
D
G
K
B
A
T

Fig. 7.—Jig for Engraving Reference Marks on Platinum Surfaces of Creep-Extensometer Units.

Since the extensometer units are mounted on the shoulders of the specimen, the measured elongation includes that which occurs in the fillets; this, however, has been shown to be quite small (see the section on Measuring the Extension). The extensometer units, shown in row *B* of Fig. 5, are of 27 per cent chromium steel machined with the inner surface tapered to fit the specimen; the nuts are of 18 per cent chromium—8 per cent nickel steel.

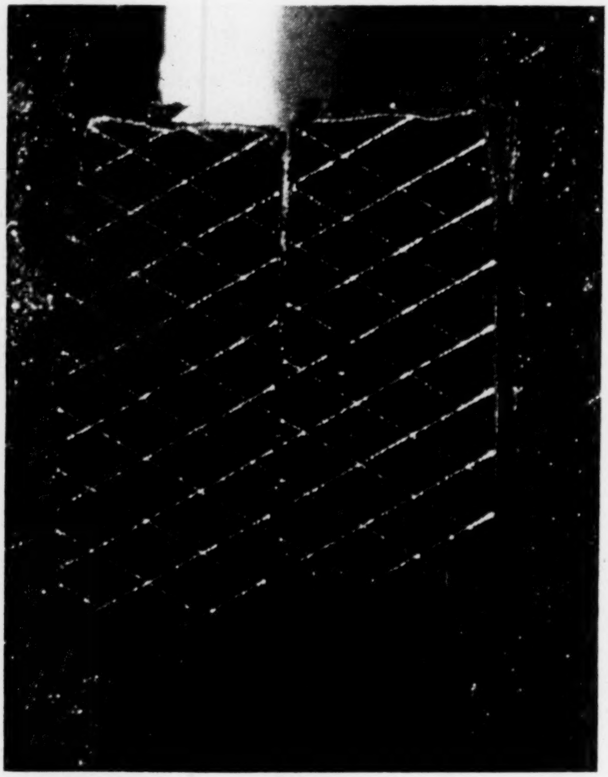


FIG. 8.—Engraved Platinum Reference Surfaces (× 40). Reduced one half in reproduction.

Preparation and Engraving of the Platinum Reference Surfaces:

In measuring with the microscope the elongation of the creep specimen, the platinum reference surfaces are magnified 100 diameters, and it is therefore necessary to have them well polished. The specimen, after being assembled to the state shown in *C* of Fig. 5, is mounted in a jig (Fig. 6), to hold the specimen assembly flat during the polishing operations. Polishing is then carried out according to standard metallographic practice with the exception that the beads need not be so free from scratches.

Engraving is done in the jig shown in Fig. 7. The specimen assembly is clamped in V-blocks *K* and placed on the table *T* against the stop screw *A* which is initially backed off so that engraving is begun at the edge of the platinum farthest away from screw *A*. Table *T* is a moving block pivoted in the center to permit rotation of 60 deg.

in the plane of its base, and is initially placed in the position shown in the photograph. The engraving tool is a 60-deg. conical diamond *D* whose horizontal position relative to the platinum surfaces *a* and *b* is adjustable through screw *B*. The engraving stroke is controlled by the screws E_1 and E_2, which limit the motion of the vertical frame *F* about point *G*. The frame *W*, pivoted at *M*, is counterbalanced for control of depth of engraving, and elevation is controlled by chain *P* over pulley *C*.

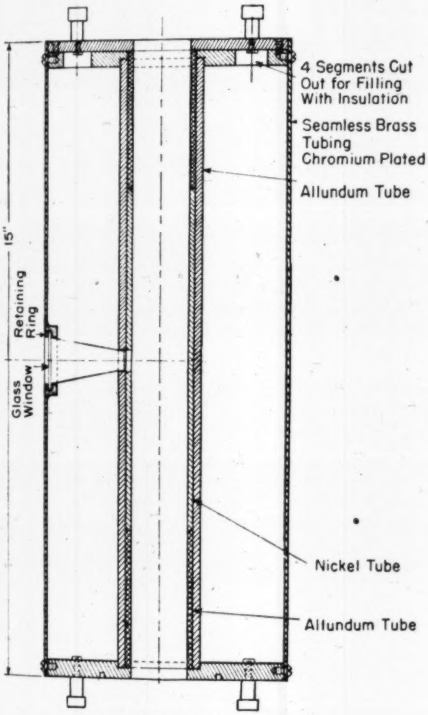

4 Segments Cut Out for Filling With Insulation
Seamless Brass Tubing Chromium Plated
Allundum Tube
15"
Retaining Ring
Glass Window
Nickel Tube
Allundum Tube

FIG. 9.—Creep Furnace.

With frame *F* forward against stop E_1, the diamond is lowered by releasing pulley *C* until the chain is slack, and then handle *H* is pushed slowly towards stop E_2. The diamond is then raised by pulley *C*, and screw *A* is turned one-half revolution to advance the specimen assembly to the position for the second engraved line. This process is repeated until the whole length of the surface has been engraved and then repeated for a second series of lines with Table *T* rotated against stop *J*. The polished surface is protected by wrapping in cotton until ready for use. The appearance of an engraved surface is shown in Fig. 8.

After preparation of the reference surfaces, the specimen assembly is mounted and aligned in the pull rods as shown in *D*, Fig. 5. A check on alignment is obtained by putting one end of the assembly in a lathe chuck and rotating it; if the specimen runs out more than $\frac{1}{8}$ in. at the other end, it is readjusted in the pullrods.

Temperature Control:

Temperature must be accurately controlled during the creep test for two reasons: first, because it exerts a great effect on creep strength; second, because the magnitude of periodically observed extension is of the same order as that produced by a change of temperature of 2 F.

The creep furnace, shown in Fig. 9, consists of heating elements, Nichrome wire wound on an Alundum tube, enclosed in a casing of chromium-plated seamless brass tubing (to minimize loss of heat by radiation). The ends, one of which is removable, are insulating Transite disks, and Sil-O-Cel No. 3 is used as thermal insulation between heating unit and casing. A sleeve of nickel inside the Alundum tube, held centrally by means of top and bottom Alundum spacers, promotes uniformity of temperature over the gage length of the specimen.

In the opening in the brass window frame of the casing, a conical Nichrome tube is fitted which extends through the heating unit tube to the inside of the furnace without touching the nickle sleeve. A circular glass disk (of microscope slide quality) is held against asbestos packing by a threaded retaining washer to shut off air currents through the window opening. The circular window frame is silver-soldered to the casing.

There are separate windings of Nichrome wire (No. 22 gage B & S) on the top and bottom halves of the tube; the spacing is governed to some extent by the temperature range in which the furnace is to be used. The ends of the windings are brought to terminals mounted on the end disks at the top and bottom. The two windings are connected in parallel, and the temperature of each adjusted independently. The maximum power required is about 750 w. for temperature up to 1500 F.

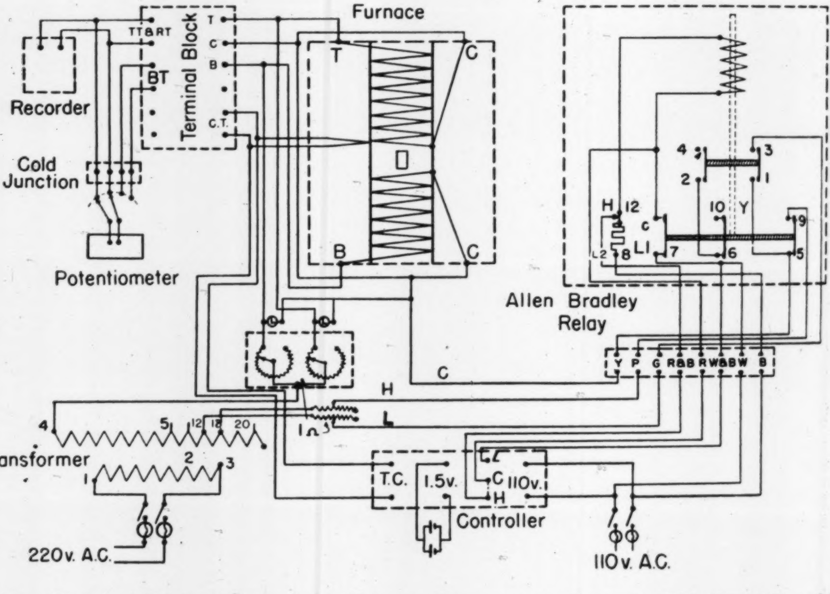


FIG. 10.—Electrical Wiring Diagram for Furnace Temperature Control. CT, TT, RT, and BT on the terminal block refer to control, top, and recording, and bottom thermocouples respectively.

The control thermocouple (No. 14 gage Chromel-Alumel) is located at the heating elements in a groove cut on the side of the Alundum tube, and is brought out through the top disk. Using a suitable controller with this couple, the specimen is maintained within ± 2 F. at temperatures up to 1100 F. and within ± 3 F. at temperatures from 1100 to 1500 F. except for unusual occurrences. The temperature uniformity over the central 3 in. of the furnace is ± 1 F. from 850 to 1100 F. and ± 2 F. from 1100 to 1500 F.

The controllers operate relays which cut in and out any two secondary taps on transformers. These transformers have 220 v. primary, and 15 taps (3 v. per tap) on the secondary, giving a possible adjustment from 30 to 75 v. A wiring diagram showing the connections from transformer to controller, relay, and furnace is shown in Fig. 10. The common tap 4 of the transformer leads to the top *T* and bottom *B* furnace windings through fine adjustment rheostats. The two other transformer taps lead through 1-ohm variable resistors to the relay, which connects one or the other to the common lead *C* to the furnace. These two taps are selected according to the furnace temperature desired. The higher voltage tap should be sufficient to maintain the test temperature about 25 F. above, and the lower one 25 F. below, the specified temperature; this limitation of the high and low voltage is an important consideration in maintaining close temperature control. Correct test temperature is established and maintained by the Chromel-Alumel control thermocouple (C. T. on the terminal block) located in the windings.

Temperature Uniformity of Test Specimens:

The checking of uniformity of temperature over the gage length of the specimen is made with five newly calibrated Chromel-Alumel thermocouples (No.. 22 gage), accurate within 0.5 F., spaced along a dummy specimen identical to the test specimen assembly which has been mounted and installed in the furnace just as for regular testing. Three thermocouples are spaced along the reduced section, the other two inserted through the pull-rods to the shoulders of the specimen (see Fig. 4(*a*)) as in a regular test. The tips of the three thermocouples spaced along the reduced section are inserted in small holes drilled into the center of the specimen and are held in place by fastening a wire around the thermocouple and specimen.

Before the furnace is balanced thermally, it is allowed to "float" on the lower power by closing the power switch while the controller switch remains off. The final temperature under this condition should be about 25 F. lower than the test temperature. Next the controller switch is closed, the controller dial shifted to the highest point, and the temperature allowed to rise to a maximum. The resistance on the high side of the secondary is then adjusted until the final temperature is about 25 F. higher than the test temperature. Finally, the controller is turned to the setting necessary to control the furnace at the test temperature. The temperature of each end of the specimen is measured, and, if necessary, adjusted by means of the fine rheostats until they are identical. When the final adjustment has been made, the temperature as indicated by each thermocouple is read. If the uniformity is not within ± 1 F. for temperatures up to 1100 F. and ± 2 F. for temperatures up to 1500 F., the furnace is rewound.

During actual test, two thermocouples are used, one through the bottom and the other through the top pull rod extending into the shoulders of the specimen. Chromel-Alumel extension wires lead to a central cold junction and reading station. The cold junction is wired to a multiple switch at a potentiometer by means of copper leads. New thermocouples are used for each creep test.

The extension leads are of the same composition as the thermocouples, Chromel and Alumel, to eliminate the need for compensating lead wires and the possibility of small errors. For the cold junction, we have installed a thermos bottle inside a refrigerator maintained only slightly above freezing temperature and accordingly need give it attention only about twice a month.

In all cases, the specimen is brought to the test temperature and maintained there for about 20 hr. (overnight) before the stress is applied. This is done by gently lowering the thrust bearing *9*, (Fig. 2) which supports the lever. At the end of test, the specimen is unloaded while still at temperature; the attendant elastic contraction is a rough measure of the elastic modulus of the metal.

Measuring the Extension:

The rate of extension in the creep test is of the order of 0.1 to 1.0 per cent per

10,000 hr. (respectively, 0.00001 to 0.0001 per cent per hour). When it is considered that this information is to be obtained in a test of relatively short duration—3 to 4 months—it is evident that sensitive and accurate measuring apparatus is necessary.

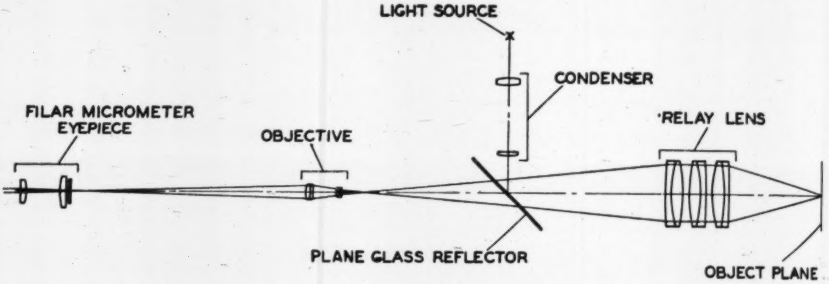

LIGHT SOURCE
CONDENSER
FILAR MICROMETER EYEPIECE
OBJECTIVE
RELAY LENS
PLANE GLASS REFLECTOR
OBJECT PLANE

FIG. 11.—Optical System of Creep Microscope.

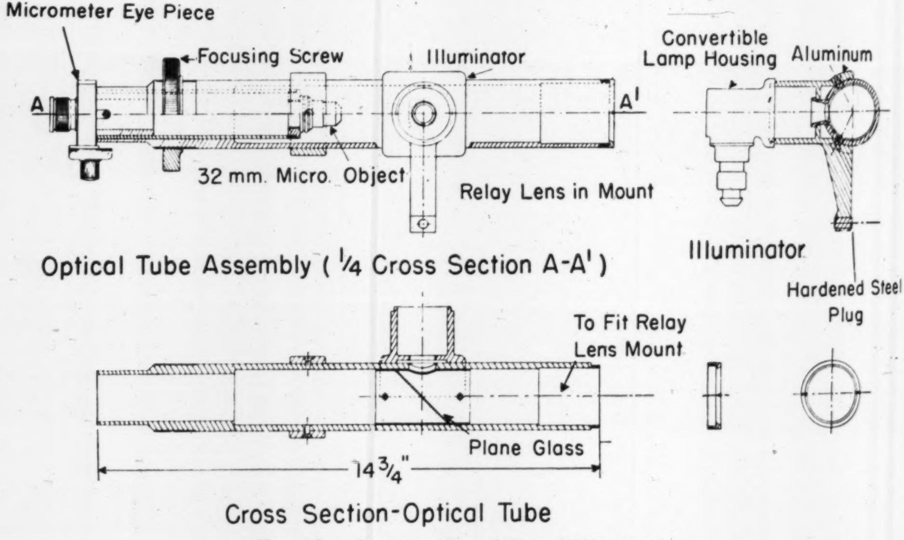

Micrometer Eye Piece
Focusing Screw
Illuminator
Convertible Lamp Housing
Aluminum
A
A'
32 mm. Micro. Object
Relay Lens in Mount
Optical Tube Assembly (1/4 Cross Section A-A')
Illuminator
Hardened Steel Plug
To Fit Relay Lens Mount
Plane Glass
14 3/4"
Cross Section-Optical Tube

FIG. 12.—Construction of Creep Microscope.

The measuring microscope used was developed in conjunction with the Bausch & Lomb Optical Co., its essential feature being a relay lens with a working distance, 70 mm, sufficient to pick up the image of the reference marks near the center of the furnace and present it to an objective of necessarily short focal length. Included in the working distance must be an added margin to avoid excessive heating of the lens. The fundamentals of the optical system are shown in Fig. 11.

The relay lens magnifies the object two diameters and projects the image so that it can be picked up by the objective of a "conventional" microscope. The total magnification of the optical system is 100; its numerical aperture is 0.25. The 2X relay lens is used with a 32-mm. objective resulting in a larger field than that obtained with a 1X relay lens and a 16-mm. objective. The filar-micrometer eyepiece has a magnification of 10. The vertical illuminator consists of a concentrated filament lamp, a condenser system, and a plane glass reflector mounted in the optical tube, construction details of which are shown in Fig. 12. A light green filter may be mounted between the condenser and the plane glass reflector to ease eyestrain.

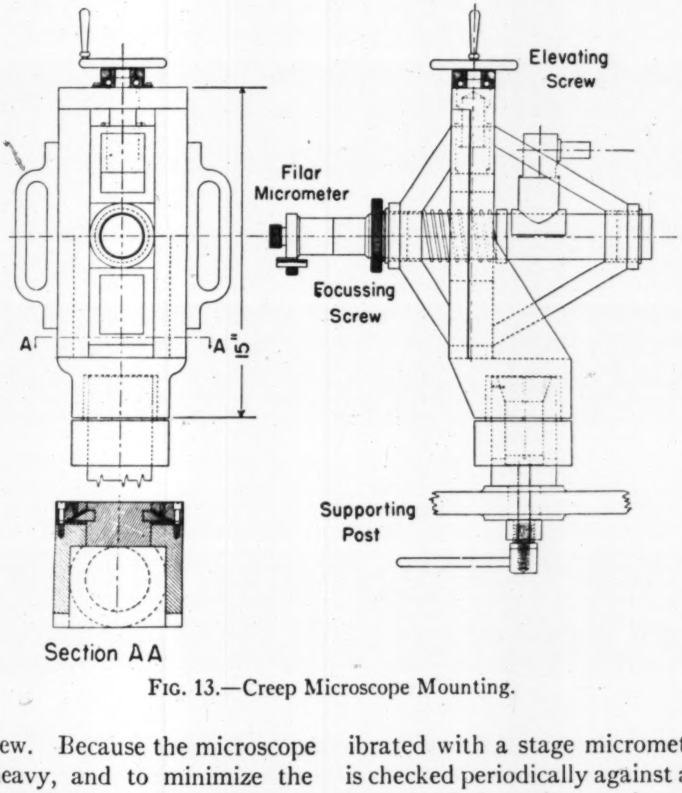


FIG. 13.—Creep Microscope Mounting.

The optical tube is fixed, as shown in Fig. 13, in a mount in which are incorporated an elevating mechanism and focusing screw. Because the microscope mount is heavy, and to minimize the danger of dropping it, we have recently constructed a mobile support, shown in Fig. 3, with which to move it from stand to stand; to facilitate moving the microscope from this support to the creep stand, it is attached over a pulley to a counterbalancing weight.

The microscope supporting post on each stand is also shown in Fig. 3. When the handle at the bottom is turned, the inner conical shaft is drawn down and expands the slotted cylinder to hold the microscope firmly. A steel ring fits over this assembly and is held in place by set screws. This ring can be raised or lowered to set the correct elevation of the microscope with respect to the furnace window, and carries a stop that may be adjusted to align the optical axis on the reference surfaces.

One filar unit on the micrometer eyepiece is equal to 0.0000398 in. as calibrated with a stage micrometer; and it is checked periodically against a standard length. Independent observers can check to one filar unit. Extension measurements are made as a rule every second day, except over weekends, when the interval is three days.

Since the extension is measured between shoulders, there was some question of an error owing to extension in the specimen fillets. Although such an error may be expected to be small, it was thought desirable to determine its real magnitude. Such a test was made by welding small platinum markers to the ends of the straight gage section, and comparing the extension, measured after test, between these marks and that as measured in the ordinary way between shoulders. With an assumed gage length of 2.3 in. for the latter method as is customary, it was determined that this method results in an extension greater by only 15 parts in 400, or about 3.5 per cent, than the actual extension over the gage length.

CREEP-RUPTURE TESTING

The Test Stand:

The creep-rupture stand is identical with the creep stand except that the miscroscope support *10* (Fig. 2) is not needed, and that the thrust bearing *9* for supporting the lever has been removed to allow for greater lever movement as required by the sometimes considerable extension of the specimen. At this position in the frame, a trough has been formed and a hydraulic jack at the base of the trough is used to support the lever arm when the specimen is not under load, to insure gentle application of the load, and finally as an effective shock absorber when the test specimen ruptures. A bracket, suspended from the two sides of the front of the frame, has been added to catch the furnace when the specimen breaks. The fall of the lever at rupture throws a knife switch to cut out the heating current to the furnace; this serves the dual rôle of shutting off the furnace and of causing the time at rupture to be recorded on the temperature recorder.

The Test Specimen:

The creep-rupture specimen is heat treated as a blank $3\frac{1}{2}$ in. long, a $\frac{1}{2}$-in. coupon taken for examination, and the remainder machined to the $\frac{1}{4}$-in. diameter specimen shown in Fig. 4(*b*). Shallow gage marks are placed at the ends of the central 1-in. section in order to measure extension after rupture. Because this specimen has the same ratio of gage length to diameter, its elongation is comparable to that of the ordinary 2-in. gage, $\frac{1}{2}$-in. diameter tension specimen. The reduced specimen size in contrast to the $\frac{1}{2}$-in. diameter creep specimen is needed to develop stress sufficiently high to cause rupture in this apparatus. The slight taper to the gage section insures that the specimen fractures near the center.[7] The specimen is mounted in adaptors which are in turn attached to the same pull rods used in the creep test.

Temperature Control:

In the creep-rupture tests, the furnace window is not needed; yet it was convenient to use the same furnace, and for the most part the same electrical equipment, as in the creep test. One change is the substitution of "Variac" type transformers for the step transformers and variable small rheostats; this substitution makes for convenience and has proved entirely satisfactory. The uniformity of temperature is checked with a dummy setup just as with the creep apparatus.

In this, as in the creep test, the specimen is brought to the test temperature and maintained there for about 20 hr. before stress is applied. This is done by lowering the lever by means of a hydraulic jack.

Measuring the Extension:

It is desirable to measure the extension occurring during a creep-rupture test even though the elapsed time to rupture is the primary concern. The extension is of much greater magnitude than in the creep test, and therefore the great sensitivity of the creep microscope is not needed. In fact, we have found it quite suitable to use the fall of the lever as a means of measuring extension. By running a fine metal chain from the lever to draw a pen along a drum rotating at constant speed, an autographic elongation-time curve of sufficient sensitivity and accuracy is obtained. A photograph of the apparatus is shown in Fig. 14. The drum, which is the standard Peters type used in the Southwark-Emery testing machine recorder, is

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[7] This practice, though undesirable, is often used in ordinary tension tests. It is particularly disappointing to have a specimen fail near the fillet in a long-time rupture test so that only a very inadequate measure of extension can be made.

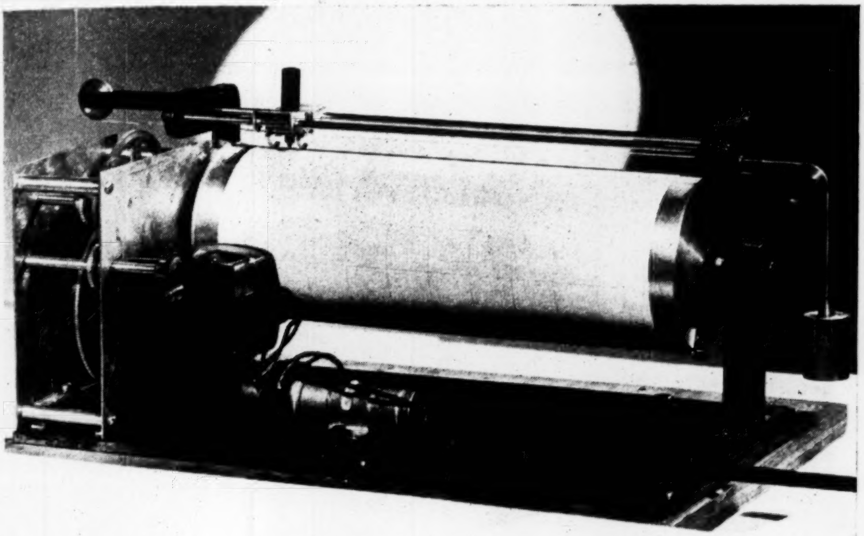

FIG. 14.—Autographic Strain-Time Recorder for Creep-Rupture Test.

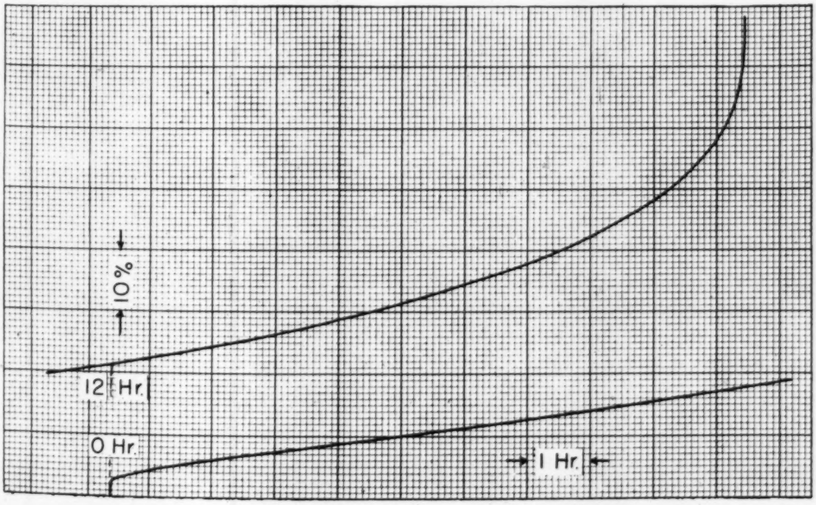

FIG. 15.—Autographic Strain-Time Record of a Creep-Rupture Test.

rotated at a rate of one or two revolutions per day. Since the chart on the drum is divided into 0.1-in. divisions, and since the effective reduced section of the test specimen is very nearly 1.25 in., it is convenient to take the motion of the lever arm at a position corresponding to a ratio of 8 to 1, for then the chart reads directly in the proportion of 1 to 10, that is, 0.1 in. on the chart corresponds to 1 per cent extension of the specimen.

A very small error is caused by extension in the specimen fillet,[8] and the final elongation as indicated on the chart differs from that measured on the fractured specimen for 1.0-in. gage length whenever local necking occurs, that is, when the deformation becomes inhomogeneously distributed along the

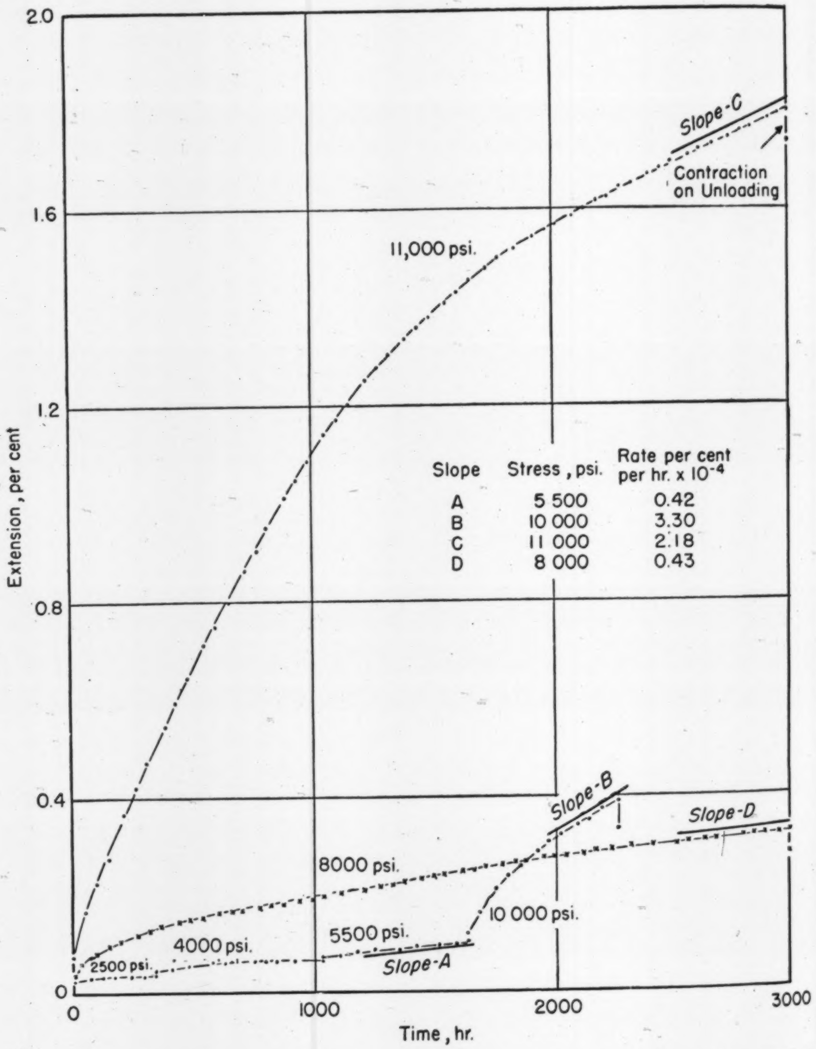

FIG. 16.—Extension-Time Plots for 18Cr–8Ni–Mo Steel (A.I.S.I. Type 316) at 1300 F.

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[8] The difference between "recorder extension" and "actual extension" as measured on a test interrupted prior to necking was about 0.4 per cent in a total of 10 per cent and part of this may have been an error in "actual extension."

specimen. In such instances the chart elongation becomes less than the actual in proportion to the extent of local necking. This is of little consequence, since the final elongation can be measured directly on the fractured specimen, and since the use made of the autographic chart is primarily to obtain the rate of extension during the "second stage," and secondarily to determine the time for the onset of the "third stage," as illustrated in Fig. 1. A representative elongation-time curve is shown in Fig. 15.

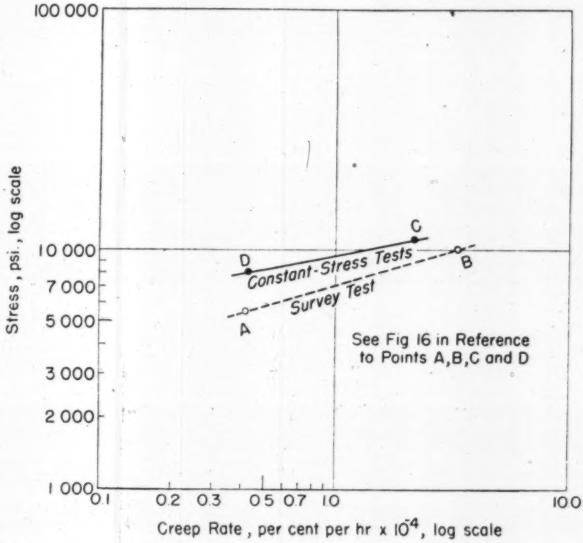

FIG. 17.—Variation of Stress with Creep Rate of 18Cr–8Ni–Mo Steel (A.I.S.I. Type 316) at 1300 F.

GENERAL PROCEDURE

Creep Test:

When setting out to determine the creep strength of a "new" material, it is desirable to run what is known as an up-step or down-step survey in order to estimate the approximate stress corresponding to the specified creep rate. As the name implies, a specimen is stressed at several successive stresses in up-step or down-step fashion, the stress being maintained at any level for the minimum time (approximately 500 hr.) needed to estimate the average rate of extension produced by that stress. Such preliminary results are not to be trusted as anything more than an approximation to the constant-stress creep rate, inasmuch as the tests are of short duration, and, with the exception of the initial step, are on material which has been plastically extended and therefore generally different from the unstrained material.[9]

The creep rate is derived graphically from the elongation-time curve with the aid of a straight-edge at 500 hr. intervals as an average over 500 hr.; that is, the creep rate at 2000 hr. is taken as the average from 1750 to 2250 hr. The minimum rate, or that of stage 2, is plotted against the stress as illustrated below to permit one to determine the stress for a specified creep rate. If a minimum rate is not attained during test, the rate at the end of test is selected, though this is evidently not a very desirable practice.

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[9] This generally unrecognized fact accounts in part, in our opinion, for the poor success of attempts at a theoretical or mathematical treatment of creep.

When a survey test is made, such as that for an 18 per cent chromium, 8 per cent nickel-molybdenum steel (A.I.S.I. type 316) at 1300 F., as plotted in Fig. 16, the rate of extension at each stress is estimated graphically with a straight-edge, and is then plotted, Fig. 17, as a function of stress.[10] From the latter plot a stress of 8000 psi. was chosen for the first constant-stress test, and, tentatively, of 5000 psi. for the second. However, upon finding that the creep rate for 8000 psi. was less than 0.1 per cent per 1000 hr., instead of greater as anticipated, the stress for the second test was changed to 11,000 psi. The elongation-time curves for these two constant-stress tests are also plotted in Fig. 16, and the creep rate at the end of each test (since each rate continuously diminished during test) in Fig. 17. From this latter plot, interpolation yields a value of 9500 psi. as the stress for a creep rate of 0.0001 per cent per hour, which, if one assumes that the creep rate remains constant, is equivalent to 1 per cent in 10,000 hr. Whether the rate does remain constant has been and is a moot point. In some tests which we have recently made it obviously does not, the third stage (Fig. 1) setting in prior to the completion of 3000-hr test even though the creep rate during the second stage is of the order of 1 per cent per 10,000 hr.; in such a case, it would appear to be foolish to report a creep strength in terms of 1 per cent in 10,000

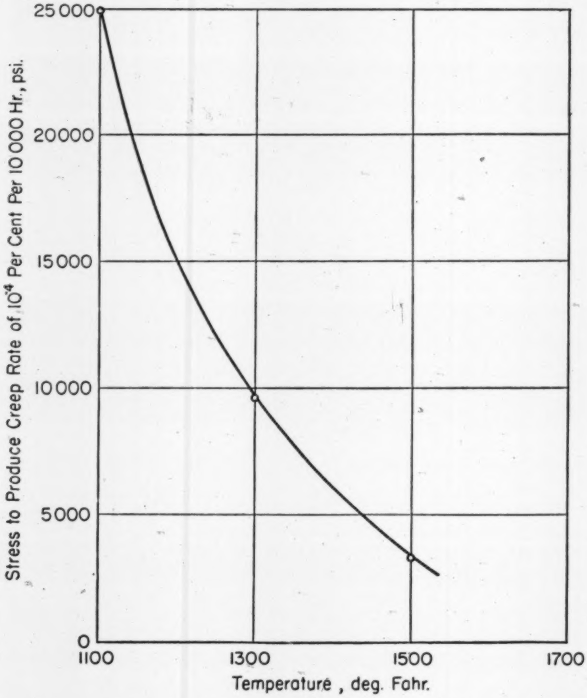

FIG. 18.—Variation of Creep Strength of 18Cr–8Ni–Mo Steel (A.I.S.I. Type 316) with Temperature

hr. However, this is not to imply that it is foolish to conduct creep tests; rather, it is necessary that the engineer using the data realize the significance of the numbers he employs in his stress formulae.

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[10] In the instance shown, the rate at the two lower stresses was too small to measure.

The log-log plot in Fig. 17 yields a straight line over a limited range of stress, but over a wide range the line is said to curve downward.[11] The untrustworthiness of the survey data is apparent from the plot; the error is often greater than in this instance. It is desirable to investigate a greater range of creep rates than indicated in Fig. 17, and, as shown below, we effectively accomplish this by combining our creep and creep-rupture data.

The pronounced effect of temperature upon creep strength is illustrated in Fig. 18; the data are for 18 per cent chromium, 8 per cent nickel-molybdenum steel (A.I.S.I. type 316), but the trend is typical.

To evaluate any microstructural changes which occur during test, the specimens tested at any one temperature are divided into two equal groups, one of which is subjected to the conventional room temperature tension test and the other examined with respect to microstructural change, hardness, and notch-impact strength. This last property is determined on two $\frac{2}{3}$- size Charpy-type specimens cut end to end from the central portion of the creep specimen,[12] which are necessarily subsize owing to the dimensions of the latter. The microstructure and hardness examinations are made on the broken notch-impact

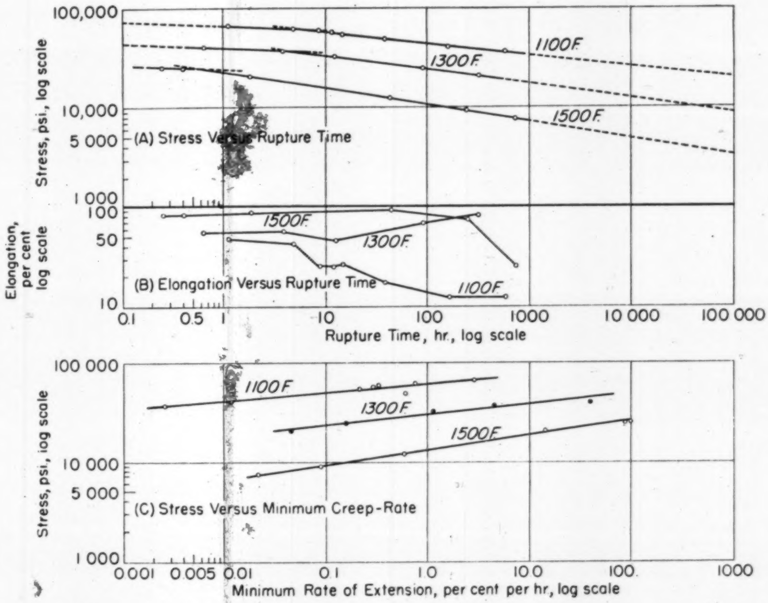

FIG. 19.—Variation of (*A*) Stress for Rupture, and (*B*) Elongation with Rupture Time, and of (*C*) Stress with Minimum Creep Rate of 18Cr-8Ni-Mo Steel (A.I.S.I. Type 316) at 1100, 1300, and 1500 F.

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[11] See A. Nadai, "The Influence of Time on Creep. The Hyperbolic Sine Creep Law," Stephen Timoshenko Anniversary Volume, The MacMillan Co., New York, N. Y., (1938).

[12] One end of each notch-impact specimen contains a portion of the thermocouple hole which extends to the shoulder of the creep specimen, but this is of no consequence.

test specimens in a region sufficiently far from that cold-worked during the test.

Creep-Rupture Test:

As pointed out earlier, the creep-rupture test is nothing more nor less than a creep test in which the stress is sufficiently high to cause rupture of the specimen while under test. A convenient indication of the stress required is given by the ordinary short-time elevated-temperature tension test. For the first of a series of creep-rupture tests at any temperature, it is generally suitable to choose a stress some 5000 to 10,000 psi. below the short-time elevated-temperature tensile strength; for the second, a stress some 10,000 to 15,000 psi. below, etc. The stresses in such a series generally plot against the time for failure, on a log-log plot, as two straight lines, as is illustrated in Fig. 19(*A*) for 18 per cent chromium, 8 per cent nickel-molybdenum steel (A.I.S.I. type 316). The break away from the initial straight line is said to coincide with a change in mode of failure from transcrystalline to intercrystalline. In a nonoxidizing atmosphere, there is reported to be no change in slope at the point where intercrystalline fracture appears, which is yet identical with that of the nonprotective atmosphere; the change in slope has been attributed to a weakening at the grain boundaries caused by oxidation.[13] The time at which intercrystalline fracture appears (the break away from the initial straight line) appears to be greater the lower the temperature. A convenient way of reporting the results of creep-rupture tests is in terms of the stress that will cause failure in some particular period of time such as 100, 1000 or 10,000 hr,; these are obtained by interpolation or extrapolation on a plot such as that in Fig. 19(*A*).

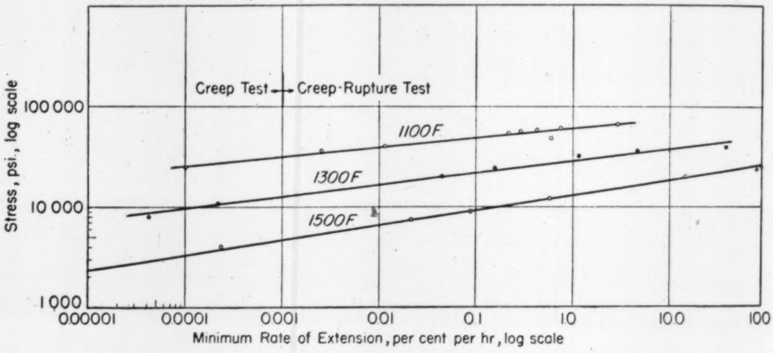

FIG. 20.—Correlation of Creep and Creep-Rupture Test Data for 18Cr-8Ni-Mo Steel (A.I.S.I. Type 316)

The elongation (and reduction of area) in a creep-rupture test is significant. For example, two specimens which under the same stress rupture in the same time cannot be considered equivalent if one has extended 5 per cent and the other 50 per cent. Ordinarily the elongation is shown on a separate plot, as in Fig. 19(*B*), or is recorded at the experimental points of the stress-time plot. Generally the elongation tends to diminish as time for rupture increases, and in an erratic fashion. The reduction of area was measured but is not given in Fig. 19(*B*) to avoid cluttering the plot.

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[13] See R. H. Thielemann and E. R. Parker, "Fracture of Steels at Elevated Temperature After Prolonged Loading," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 135, p. 559 (1939).

The rate of extension during the second stage of the time-extension curve has been determined directly from the autographic curves obtained during test and plotted against the stress in Fig 19.(*C*). A surprisingly straight line is obtained on the log-log plot.

To illustrate further the relation between the creep and creep-rupture tests, Fig. 20 has been prepared. The straight-line logarithmic relationship between stress and extension rate extends throughout the whole range studied, and the creep-rupture data are clearly continuous with the creep-test data. We have observed similar agreement in all instances in which we have made both creep and creep-rupture tests.

We have been conducting creep-rupture tests for only a relatively short time, and have not yet developed a standard program of after-test examination. We intend at first to spend considerable time studying the type of fracture, intergranular or transgranular, and the conditions under which each occurs. Our preliminary results, it might be noted, indicate that some of the characteristics attributed to the break in the log-log stress *versus* rupture-time plot are not always justified.

Use of the Data:

Both creep strength and rupture strength as employed in the design of a structure for long-time service at elevated temperature necessarily involve extrapolation. The rupture strength can be extrapolated with a fair degree of confidence owing to the well-defined relation between stress and rupture-time; but the creep strength with less confidence, owing primarily to uncertainty as to when the extension-time curve will enter stage 3 (Fig. 1).[14] Because of this latter fact, it is well to consider also the rupture strength even when the amount of distortion is the primary concern, for doing so will at least insure that actual rupture does not occur. As an example, we might take the data for 18 per cent chromium, 8 per cent nickel-molybdenum steel (A.I.S.I. type 316) presented here. At 1300 F., the stress to produce a deformation of 1 per cent in 10,000 hr. (0.0001 per cent per hour) is 9500 psi. (Fig. 17), and let this be the stress which we choose, on the basis of the permissible distortion, for a structure which is to last 10,000 hr.[15] We then extrapolate the 1300 F. stress versus rupture-time curve of Fig. 19(*A*) to the stress of 9500 psi. and find that the time for rupture at this stress is 60,000 hr. Thus the design stress appears to be relatively conservative with respect to whether or not rupture occurs. In an actual design, a factor of safety will, of course be applied.

When the distortion occurring in a structure used at elevated temperature is of no consequence, design can be based on rupture strength alone, except in so far as one might wish to use the distortion as a measure of impending rupture.

Acknowledgment:

Of those who aided in the development of this apparatus and procedures, we wish particularly to acknowledge the contributions of Messrs. J. W. Bain, R. F. Campbell, Q. Henderson, and E. L. Roff.

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[14] In the near future, we hope to present data which will define the relation between stress and time for beginning of stage 3.

[15] It should be recognized that in the example cited, the extension at the end of 10,000 hr. will be greater than 1 per cent by (*a*) the elastic extension corresponding to the stress employed, and (*b*) the amount by which the plastic extension occurring in stage 1 is greater than if there had been no such stage, that is, than if stage 2 had started at zero time.

DISCUSSION

Mr. P. G. McVetty.[1]—The authors have mentioned the need for accurate temperature control on account of the effect of temperature upon creep strength and the similarity between thermal expansion and creep. Another important reason must be considered in tests at high temperatures. It has been common practice to base designs on the maximum temperature expected in service. This procedure follows the assumption that the effects of any variations of temperature in service are less severe than continuous exposure to the maximum temperature. This assumption has been found satisfactory in many cases but there is evidence of increasing importance of temperature variations with increasing temperature. For example, Fellows, Cook and Avery reported[2] a considerably greater creep rate in a test having a cyclic temperature variation between 1790 and 1810 F. than in a similar test at a constant temperature of 1810 F. They found also in a test at 1800 F. an increase in creep rate of about 60 per cent after a power interruption of 10 min. These factors must be considered when service applications involve temperature variations and available test data were obtained under constant temperature conditions.

Mr. W. C. Ellis.[3]—In the presentation of data for creep rates of lead alloys we have generally used a semilog plot of creep rate as a function of stress. In a number of instances the data have fallen on two intersecting straight lines of different slope. This we have interpreted as indicating a creep mechanism at low stresses different from that at high stresses.

Creep data have been presented in today's session plotted on a log-log basis in which in many instances straight line plots were not obtained. I should like to ask the authors of this paper whether from fundamental considerations of the mechanism of creep one should expect a linear relation between creep rate and stress on a log-log plot?

Mr. Perry R. Cassidy.[4]—I had the pleasure and opportunity a short time ago of a personally conducted inspection with Messrs. Aborn[5] and Smith of this excellent equipment.

Very modestly, as usual, Mr. Aborn did not mention the fact that all of this equipment was produced by his own laboratory, and it is beautiful equipment.

There is one question that I should like to ask. The creep equipment which we have at M.I.T. was installed some twenty-odd years ago and has a very much longer gage length and a larger diameter, particularly, in regard to the stress rupture specimens that the authors use. Has anyone noticed or correlated the effect of variation in gage length and diameter of the specimen on the results of creep and creep rupture?

Mr. Ward F. Simmons[6] (*by letter*).—

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[1] Manager, Strength of Materials Section, Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

[2] J. A. Fellows, Earnshaw Cook, and H. S. Avery, "Precision in Creep Testing," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Iron and Steel Div., Vol. 150, p. 360 (1942).

[3] Bell Telephone Laboratories, Inc., Murray Hill, N. J.

[4] Executive Assistant, The Babcock & Wilcox Co., Barberton, Ohio.

[5] Mr. Aborn presented the paper for the authors.

[6] Research Engineer, Battelle Memorial Inst., Columbus, Ohio.

This paper is a good description of the creep and creep-rupture apparatus and the testing techniques used at the Research Laboratory of the United States Steel Corp. "Creep-Rupture" is a much better descriptive name for these tests than the more commonly used "Stress-Rupture," especially for tests where time-deformation data are obtained.

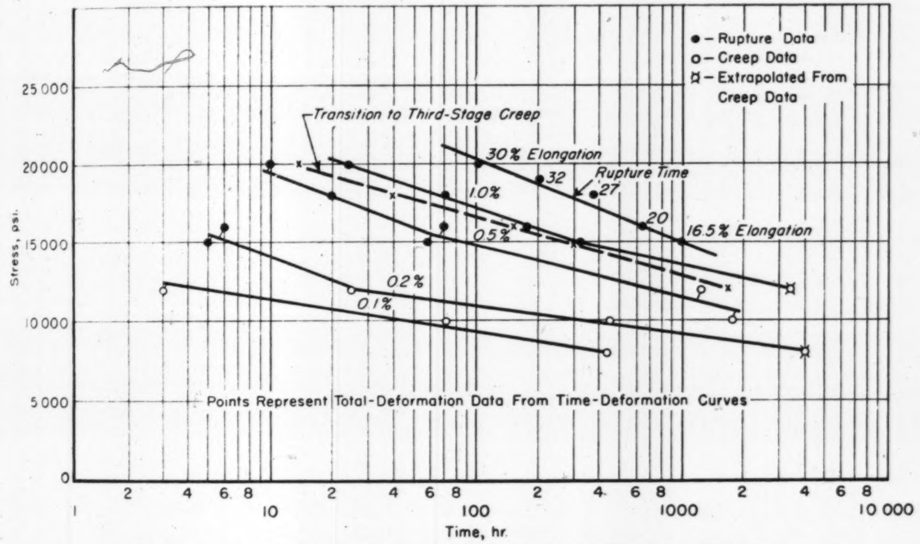

FIG. 21.—Design Curves at 1500 F. for S-590 Alloy Disk, NR-74B.
Heat Treatment: Water-quenched after 3½ hr. at 2300 F. plus 16 hr. at 1400 F.

In the section dealing with creep-rupture testing, the authors describe their method of measuring the extension occurring in a rupture test by utilizing the fall of the lever arm to represent extension in the test section of the specimen. I think that a word of caution is in order here. In our creep laboratory at Battelle Memorial Institute we attempted to use 0.0001-in. dial gages actuated by arms on the pulling adapters just outside of the test furnace, to measure extension in rupture tests. In these tests the extension was also measured by our creep method, which is similar to that used by the authors. Acceptable agreement between minimum creep rates determined by the two methods was obtained on only 30 per cent of the tests, and in all tests the rates obtained from the dial-gage measurements were higher than those measured by creep methods. Creep in the threads of the specimen and pulling adapters was probably responsible for the higher rates obtained from the dial-gage measurements.

The authors have shown several methods of presenting creep and creep-rupture data. Another method is shown in the accompanying Fig. 21, which is a typical design curve. Design curves are made by plotting the times required to reach certain specified total deformation values. Both creep and creep-rupture data are used, and the rupture time, and the time for the beginning of third-stage creep are usually given. Data of this type require that accurate total deformation measurements be obtained on creep-rupture tests as well as on the creep tests.

MESSRS. G. V. SMITH, W. G. BENZ, AND R. F. MILLER (*authors' closure by letter*).—The possibility of an influence of a variable temperature on creep strength, as mentioned by Mr. McVetty, is of considerable practical interest. We have made a few preliminary tests of the influence of cycling temperature which, however, seem to show little or no effect. The range of cycling employed was 100 F. and the maximum temperature tested was 1550 F., somewhat below that employed in the reference cited by McVetty. Of course, it is to be expected that the creep rate in a test with cycling temperature should be somewhat greater than that in a constant temperature test corresponding to the mean temperature of the cycling test, in-as-much as creep rate increases more than proportionately with temperature. When this is considered, cycling temperature, within the limits studied in our Laboratory, seems to exert little if any influence. As yet we have made only a few tests and it is possible that further tests, now under way, may reveal an effect under some conditions.

In reply to Mr. Ellis, we have employed the log-log plot, represented mathematically by a power relation, because it is most widely used and fits the data reasonably well. Theoretical treatments, which cannot be reviewed here, however, seem to indicate that a hyperbolic sine relation is more proper, and as mentioned in the paper a few experimental data seem to support this conclusion. We are quite interested to learn of the observation of Mr. Ellis that semi-log plotting may result in two straight lines of different slopes.

We appreciate Mr. Cassidy's complimentary remarks. In regard to the length of specimen, we should regard this as immaterial. Of course with increasing gage length, the unit strain is increasingly magnified, but at the same time, the problem of temperature uniformity and control is more serious. The diameter of the specimen is of some interest in creep to rupture tests, however, according to the results of Thielemann and Parker[7] who observed that when intergranular fracture occurs, the curve of log stress *versus* log rupture-time breaks from its original slope in an oxidizing atmosphere, but not in an inert atmosphere, and that in the former case the time for rupture beyond this transition point varies somewhat with the specimen size.

Mr. Simmons cautions that extension measurement outside the furnace may lead to some error owing to creep in the specimen threads and adaptors. We have not encountered this difficulty. Presumably it would be readily detected, if of consequential magnitude, when attempting to fit the next test specimen into the adaptor. The so-called "design curve" submitted by Mr. Simmons is a valuable method of presentation in some cases, although somewhat laborious, and has been effectively used by many investigators in reporting creep results, particularly during the war for such applications as in gas turbines. In other and probably more important cases, in which interest centers on small strains and on extrapolation to long-time behavior, the "design curve" does not appear to be a suitable means of presentation in some instances as judged by some plots shown in the literature, as well as that submitted by Mr. Simmons.

It may be of interest to state that since preparing our paper, we have been able to obtain a reasonably fair value of total creep in our creep to rupture tests by correcting for the strain occurring other than in the specimen immediately on loading. During test, only the specimen undergoes significant creep.

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[7] R. H. Thielemann and E. R. Parker, "Fracture of Steels at Elevated Temperatures After Prolonged Loading," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 135, pp. 559–582 (1939).

THE PROGRESS OF FAILURE IN METALS AS TRACED BY CHANGES IN MAGNETIC AND ELECTRICAL PROPERTIES*

BY P. E. CAVANAGH[1]

SYNOPSIS

The relative changes in magnetic and eddy-current losses during normal endurance tests in rotating beam machine have been determined for six metals at loads above and below endurance limit. Easily detectable changes in these losses occur at loads above the endurance limit. In certain special instances this fact may be used to give a useful method of predicting that a particular metal will fail in normal service.

A series of endurance tests was run at various test speeds to determine whether changes in magnetic and eddy current losses would indicate partial suppression of normal slip at such test speeds.

The changes in magnetic and electrical properties accompanying the development of failure during rotating-beam endurance tests have been compared for six different metals and alloys. This general survey was suggested by some interesting and practical results obtained by use of the test method to detect the beginning of plastic deformation in metals.

The physical basis for the correlation existing between elastic and plastic deformation in metals caused by an external stress and the accompanying magnetic and electrical effects is well known to physicists. The present state of the domain theory of ferromagnetism provides the metallurgist with a practical model to use in visualizing the manner in which lattice distortions caused by internal or external stress can change the magnetic state of ferromagnetic metal (**1, 2, 3, 4, 5, 6, 7**).[2] There are objections to the present treatments of domain models (**8**) but there is no doubt that present theory provides a practical tool which may be used to calculate the magnetic properties of metals under given conditions and to predict their behavior under the influence of stress (**9**).

The proof of the dependence of the free crystal energy, under small homogeneous strains, on the direction of magnetization within the crystal has led to experimental application of the determination of magnetic properties to discover the mechanical state of strain in the elastic range in polycrystalline metals (**10, 11, 12, 13, 14, 15**). In a ferromagnetic metal some of the magnetic properties are determined by the constraint placed on the free "movement" and interaction of magnetic domains by stresses, crystal energy, and thermal agitation. These structure-sensitive magnetic properties such as the initial permeability and remanence can be radically altered by small changes in externally applied stress.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] President, Cyclograph Services Limited, Toronto, Ont., Canada and Research Fellow, Ontario Research Foundation.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 646.

Internal stresses set up by plastic deformation modify the ease and mode of movement of domains under the influence of an external magnetic field or external stress. The resulting correlation between certain magnetic properties and the progress of plastic deformation has been investigated by several methods (**16, 17, 18, 19**).

In a nonmagnetic metal the change in electrical resistance due to plastic deformation alone is very nearly a linear function of the plastic deformation and has been used to give useful information about the state of strain (**20**).

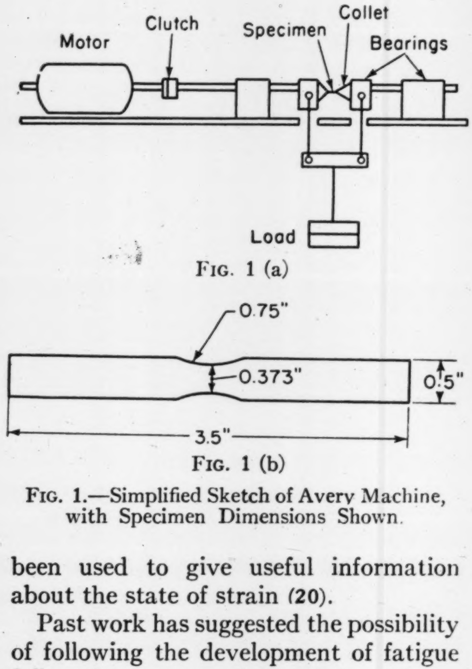

FIG. 1 (a)

FIG. 1 (b)

FIG. 1.—Simplified Sketch of Avery Machine, with Specimen Dimensions Shown.

Past work has suggested the possibility of following the development of fatigue failure by some method of determining the accompanying changes in magnetic and electrical properties (**21, 22, 23, 24, 25**). The present investigation was for the purpose of making a general survey of the usefulness of such a method and estimating the limitations of practical applications.

TEST PROCEDURES

All tests were performed with an Avery rotating-beam fatigue machine on standard specimens (Fig. 1). A Du Mont Cyclograph was used to determine and record changes in magnetic and electric properties of the samples.

Descriptions of the Cyclograph may be found in the literature (**26**). This instrument is essentially an extremely sensitive oscillator. The test coil which is placed about the specimen while an endurance test is in progress is part of the oscillatory circuit. The readings of the Cyclograph are a function of the losses in the specimen. For nonmagnetic materials these losses are due to eddy currents which are a function of the resistivity of the specimen. For ferrous materials the losses consist of hysteresis and eddy-current losses, which are a function of several factors, including the

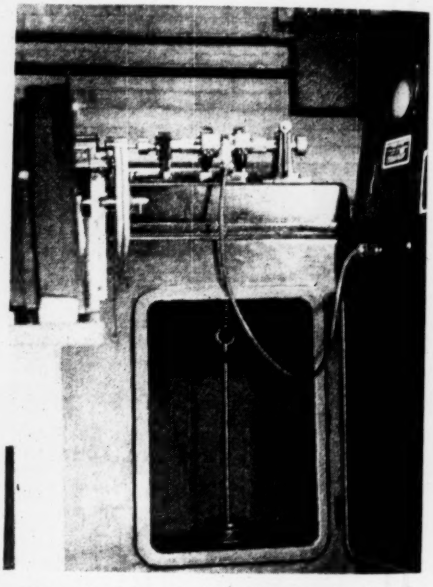

FIG. 2.—Test Setup Showing Cyclograph, Avery Fatigue Machine and Recorder. Cyclograph test coil is in place around test specimen.

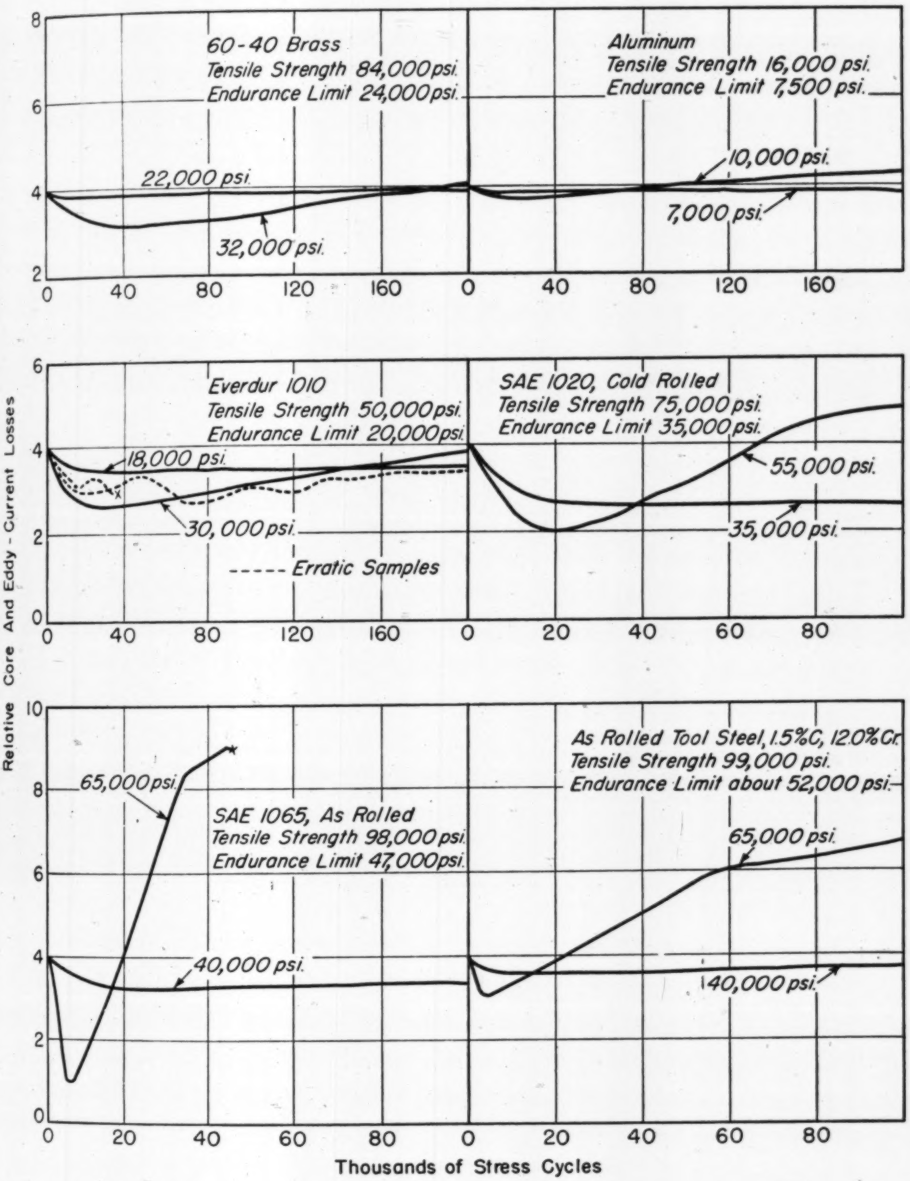

FIG. 3.—Variations in Core and Eddy Current Losses Recorded During Rotating-Beam Endurance Tests at Loads Above and Below the Endurance Limits.

permeability and resistivity of the materials under test. These losses decrease the oscillator output. Suitable circuits provide a d-c. signal proportional to magnetic and eddy-current losses which can be recorded on a 1-mil Esterline-

Angus recording milliammeter. The test setup is shown in Fig. 2. The recorder paper is mechanically driven from the fatigue machine so that paper travel is proportional to cycles of stress.

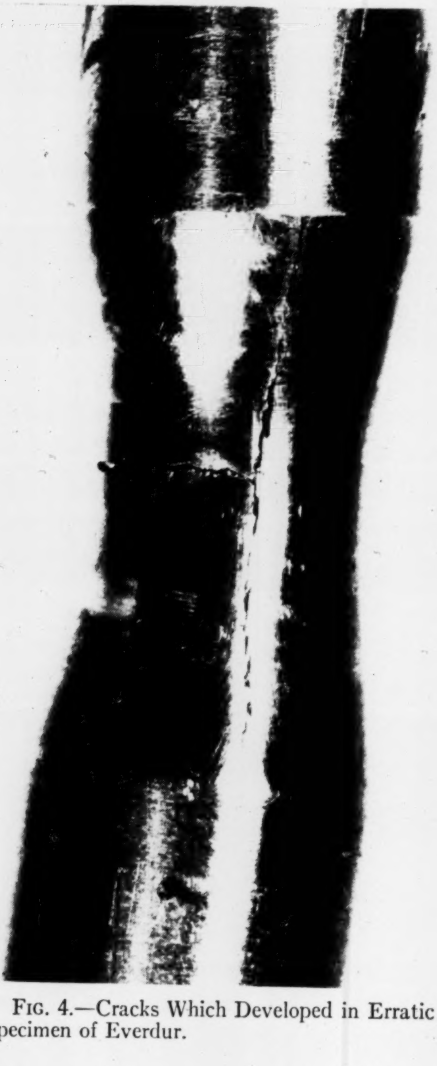

FIG. 4.—Cracks Which Developed in Erratic Specimen of Everdur.

The test frequency used in all these experiments was 5000 cycles. The field in the Cyclograph test coil is weak—of the order of 1 oersted. Under these conditions flux penetration in the annealed S.A.E. 1025 steel samples, for example, is about 0.025 in.

Effects of Fatigue Tests at Ordinary Speeds:

The first series of experiments on ferrous and non-ferrous metals was run at two test stresses. The loads chosen were about 95 and 130 per cent of the endurance limit for each metal.

At the maximum practical operating sensitivity of the Cyclograph no continuing change in magnetic and eddy-current losses could be detected when specimens were run at 95 per cent of the endurance limit. This confirms previous results obtained on various types of fatigue machines. In no instance can any further change in these losses be detected after an initial small change in the first portion of the test if the load is below the endurance limit.

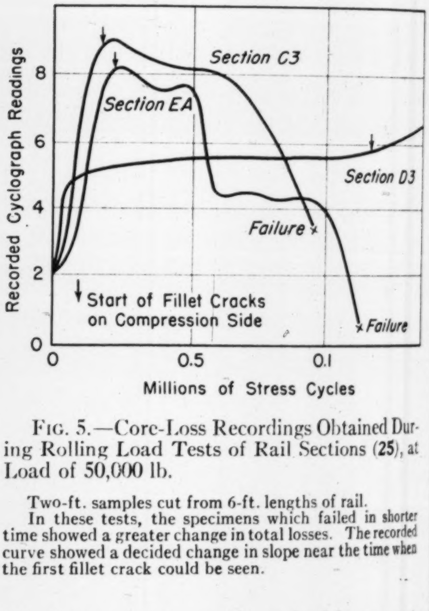

FIG. 5.—Core-Loss Recordings Obtained During Rolling Load Tests of Rail Sections (**25**), at Load of 50,000 lb.

Two-ft. samples cut from 6-ft. lengths of rail.

In these tests, the specimens which failed in shorter time showed a greater change in total losses. The recorded curve showed a decided change in slope near the time when the first fillet crack could be seen.

The recordings obtained at loads above and below the endurance limits are shown in Fig. 3. Three samples of each metal were run. The only purpose was to establish the form of the recorded curves of core-loss changes and not to obtain any quantitative data.

The curves for two of the Everdur specimens showed unexpected irregularities. When these specimens failed, a longitudinal crack appeared before the normal circumferential crack (Fig. 4). Apparently a longitudinal seam was present in the original bar stock. The resulting erratic eddy-current loss changes were produced by the abnormal manner of stress distribution and failure.

Easily detectable changes in magnetic and eddy-current losses do occur during failure at stresses above the endurance limit. Figure 5 illustrates typical results obtained during normal rolling load endurance tests of rail sections at a load of 50,000 lb. A previous investigation (23) established that the major portion of these changes is due to plastic deformation accompanying the development of failure. Results were obtained which showed that in some special instances changes in core and eddy-current losses occurred which were attributable to fatigue damage as measured by further endurance tests at a different load. It was definitely proven, however, that there was no possibility of correlation with fatigue damage alone under normal endurance test conditions.

TABLE I.—PROPERTIES OF ANNEALED S.A.E. 1025 STEEL.

0.24 per cent carbon and 0.52 per cent manganese

Yield stress, psi	42 500
Maximum stress, psi	62 000
Elongation, per cent	23
Reduction of area, per cent	60
Endurance limit, psi	29 000

Effects of Fatigue Tests at High Speeds:

Some work by W. A. Wood (27) at the National Physical Laboratories in England indicated that it is possible to run a rotating-beam endurance test at high enough speed so that normal plastic deformation is suppressed in some metals. If the rate of propagation of a dislocation is relatively slow, it is conceivable that a rapid enough application of high stress would not allow sufficient time for normal slip to occur. Some significant change in the crystal lattice must take place under such conditions because failure occurs in about the same number of stress cycles as at slower testing speeds. Fatigue damage is being produced with little or no accompanying slip. Whether or not this damage factor is normal fatigue damage, a high-speed test which produces this condition would allow elimination of the effects of normal cold working on core and eddy-current losses during fatigue failure.

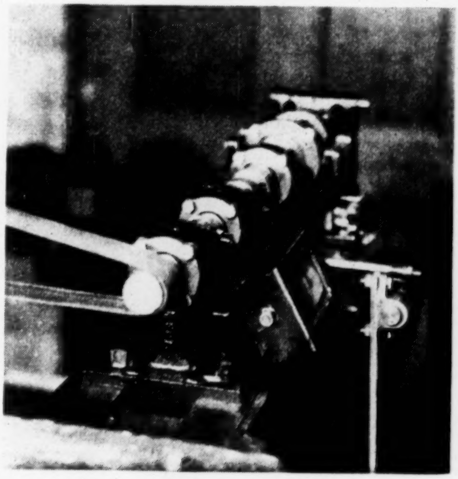

FIG. 6.—Belt Drive for High-Speed Tests. The gear train for driving recorder paper is also shown on the right.

From another aspect this type of test could give some useful fundamental information about the behavior of metals during plastic deformation. If tests on a metal were run at higher and higher speeds at a load above the yield point

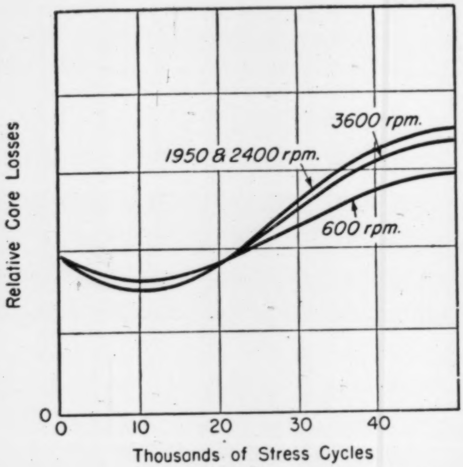

FIG. 7.—Core-Loss Variations During Endurance Tests at 38,000 psi. on S.A.E. 1025 Steel. Test speeds in rpm. are shown for each recording.

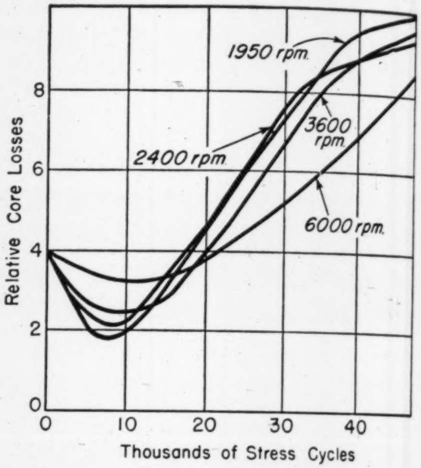

FIG. 8.—Core-Loss Variations During Endurance Tests at 50,000 psi. on S.A.E. 1025 Steel. Test speeds in rpm. are shown for each recording.

until core and eddy-current losses, or X-ray examination, showed that no slip was taking place, an approximation to the rate of propagation of slip could be obtained.

A series of experiments was undertaken on fully annealed S.A.E. 1025 steel to try to achieve failure without normal previous cold working of the metal. The properties of the steel used are listed in Table I.

The Avery fatigue machine was fitted with a belt drive as shown in Fig. 6. The speed of rotation could be varied from 1900 to 10,000 rpm. by use of suitable pulleys. Unfortunately, excessive vibration appeared at the higher speeds with all but a few specimens due to slight

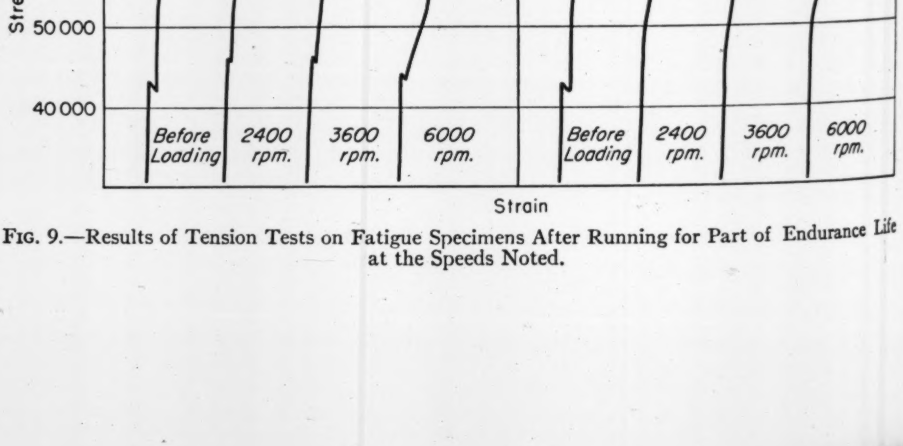

FIG. 9.—Results of Tension Tests on Fatigue Specimens After Running for Part of Endurance Life at the Speeds Noted.

TABLE II.—ENDURANCE LIFE ON SAMPLES OF S.A.E. 1025 ANNEALED STEEL RUN AT VARIOUS TEST SPEEDS.

Results are the average of three samples at each speed.

Load, psi.	Cycles at 1950 rpm.	Cycles at 2400 rpm.	Cycles at 3600 rpm.	Cycles at 6000 rpm.
50 000	137 500	135 000	129 800	134 200

eccentricity of the chucks and to bearing irregularities. Only a few satisfactory tests were run at the high speeds, but the results were sufficiently interesting to lead to further work. In all tests the specimen was placed in the chucks and loaded to 2000 psi. The machine was started and brought up to test speed and then checked for excessive vibration of the specimen. The load was then increased to the test value by very carefully adding 2000 psi. at a time. The duration of the test was determined from the time when the applied load reached the test value.

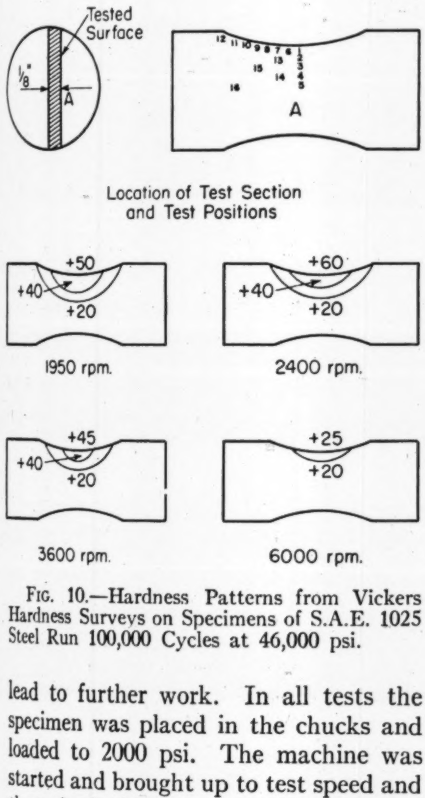

FIG. 10.—Hardness Patterns from Vickers Hardness Surveys on Specimens of S.A.E. 1025 Steel Run 100,000 Cycles at 46,000 psi.

The recorded core-loss variations for tests above the endurance limit at several test speeds are shown in Figs. 7 and 8. At the higher test load of 50,000 psi., the recorded curve at 6000 rpm. departs further from the normal curve than is the case with a lower test load of 38,000 psi. In both instances the position of the 6000 rpm. curve with respect to curves for lower speeds indicates that less plastic deformation occurs at 6000 rpm.

The endurance life at 50,000 psi. was determined at four test speeds. No significant change was produced by varying the speed of rotation (Table II).

TABLE III.—HARDNESS SURVEYS ON SECTIONS OF SAMPLES RUN AT VARIOUS TEST SPEEDS AT A LOAD OF 46,000 PSI. FOR 100,000 CYCLES.

Hardness Test Location[a]	Vickers Hardness Numbers for Test Speeds Indicated			
	1950 rpm.	2400 rpm.	3600 rpm.	6000 rpm.
1	199	188	178	154
2	192	188	172	136
3	188	170	158	136
4	166	156	139	133
5	153	149	141	133
6	192	198	185	158
7	188	191	179	156
8	183	185	173	156
9	172	175	160	136
10	166	166	151	131
11	161	161	143	131
12	149	144	141	131
13	183	166	168	133
14	161	156	159	133
15	150	148	141	131
16	149	141	141	133

[a] Hardness readings taken at locations shown in Fig. 10 with Vickers 5-kg. load.

Tests were next run for a portion of the endurance life at 43,000 and 50,000 psi. The specimens were then removed from the fatigue machine and subjected to tension and hardness tests.

Tension tests were first run on four fatigue specimens which had not been subjected to the fatigue test in order to obtain the original values for yield stress and ultimate stress. Results obtained on fatigue specimens pulled after running under the same load at various speeds are shown in Fig. 9.

Tension tests showed no change in the value of the new yield stress established by this test procedure when the test speed was increased.

Further specimens were run at four test speeds for 100,000 cycles at a load of 46,000 psi., then sectioned as shown in Fig. 10. Hardness surveys were taken on the exposed cross-section. A Vickers hardness tester with a 5-kg. load was used for these tests. Hardness values at the locations shown in Fig. 10 are listed in Table III.

Hardness patterns on the cross-sections are illustrated in Fig. 10. The maximum increase in Vickers hardness above the original hardness of each specimen is noted at the surface. Areas showing more than 40 points and more than 20 points increase in Vickers hardness are also marked. A decrease in the work-hardened area is shown at 6000 rpm.

CONCLUSIONS

The work done in this field to date illustrates that the magnetic and eddy-current losses will trace the development of failure at loads above the endurance limit in the ferrous and non-ferrous metals investigated. No change in such losses can be detected if failure is not developing.

These facts indicate a practical use of the method in examination of metal parts in normal use under operating loads to determine whether they are beginning to fail. A recording taken over a length of time during normal operation should show no change in core, or eddy-current, losses at constant load if the part is to last indefinitely.

There is no possibility of estimating remaining endurance life by this method.

No significant difference in endurance life was produced by increasing the speed of testing, although the amount of slip in the metal was materially reduced.

According to Wood's work (27), application of cyclic stresses at a sufficiently high speed to suppress normal plastic deformation will still develop internal strains which produce permanently raised yield points and hardness and which bring about fatigue failure in about the same number of cycles as slower speeds of testing.

It appears that the core loss method can be used to detect this condition in the steel tested. If the amount of plastic deformation, with accompanying crystallite formation, is reduced in an endurance test by increasing the speed of cyclic loading, the recorded core-loss curve will depart from its normal position. A maximum change of position of this curve would be reached if all normal slip could be suppressed. These facts indicate the possibility of utilizing this method to estimate the rate of propagation of slip in a metal.

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DISCUSSION

Mr. R. C. A. Thurston[1] (*presented in written form*).—This further contribution by Mr. Cavanagh to the study of nondestructive testing and fatigue phenomena has indicated the applicability of his particular method to a diversity of materials, both ferrous and non-ferrous. The difference in core loss recordings in safe and unsafe fatigue ranges under rotating bending conditions is most marked, and though the Cyclograph may not be capable of estimating remaining endurance life or detecting the extent of fatigue damage, its possibilities in the field of fatigue testing alone are numerous. At the Bureau of Mines in Ottawa we have on loan one of the Du Mont Cyclographs. This instrument is being used, at present, to investigate the changes in normalized S.A.E. 1035 steel under reversed direct stresses in a 2000-lb. Sonntag fatigue machine, speed 1800 rpm. Only a limited number of tests has been carried out to date, but they show general agreement with those of the author in the unsafe stress ranges.

The results obtained with the Cyclograph in the fatigue tests at various speeds are particularly interesting. Wood and Thorpe,[2] using brass specimens under reversed direct stresses, have shown that the dispersal of grains into widely oriented crystallites, the characteristic of the yield point under static or slow cyclic stress, is entirely suppressed under the same stress ranges when applied at higher frequency (2200 cycles per min.). The persistence of this suppression was indicated by subsequent mechanical tests. Similar observations were made by the author on annealed S.A.E. 1025 steel, although the conditions of dynamic stressing were entirely different, the stress across the section being non-uniform.

The core-loss curves for different speeds at stresses above and below the yield point, both show a progressive trend, the former being more marked. I should like to ask if there is any possibility of a temperature effect here? Oberg and Johnson[3] have shown with stainless steel, under similar conditions, that the temperature rise in the test section increases with the speed of rotation and the applied load. At the endurance limit of their material, 70,000 psi. and a speed of 10,600 rpm., they reported a temperature of about 180 F.

Referring to the author's remarks on the rate of propagation of slip, and the effect of speed of cyclic loading, Jenkin and Lehmann[4] carried out some fatigue tests in 1929, employing a special type of pneumatic machine capable of producing frequencies up to 20,000 cycles per sec. Tests on several materials showed that the fatigue strength rose to a maximum at 10,000 to 20,000 cycles per sec. and then sometimes diminished; increases up to 60 per cent were observed. Hardness tests on fractured specimens also showed an increase with testing speed. The author, however, has reported a decrease in hardness and the work-hardened area for unbroken specimens as the speed is increased. It would seem that this point might repay a little further investigation.

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[1] Metallurgical Engineer, Department of Mines and Resources, Mines and Geology Branch, Mineral Dressing and Metallurgy Div., Ottawa, Ontario, Canada.

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In conclusion, the author has stated in his paper that "there was no possibility of correlation with fatigue damage alone under normal endurance test conditions." It would be interesting to know whether the Cyclograph has been applied to the case of a specimen damaged by overstressing, and subsequently healed by progressively increasing cyclic understress.

MR. H. F. MOORE.[5]—As one who has tried with very indifferent success some half dozen tests for short-time fatigue strength I always look with hope toward the work of other investigators who are trying still other ways of making short-time tests.

As I understand the paper, the author reports that by making fatigue tests up to something like 100,000 cycles of stress he can determine with some accuracy whether the endurance limit for indefinitely large number of cycles of stress has been reached. Do I understand him correctly, and specifically can he determine whether such tests can distinguish between plastic action and the formation of a spreading fatigue crack?

If that can be done it would make it possible to reduce the time required for fatigue testing very appreciably—perhaps by 50 per cent. Especially would I ask whether he gets good results for tests of non-ferrous metals.

MR. JOHN A. BENNETT[6] (*by letter*).—I should like to ask two questions regarding Mr. Cavanagh's very interesting paper. First, in regard to the appearance of the fracture in the Everdur specimens which failed with a longitudinal crack. Did the transverse crack start from a point on the seam? We have had one or two service failures submitted which were somewhat similar to this, and it would be interesting to compare them with these laboratory specimens where the stress was known to be longitudinal.

Only one of the curves in Figs. 3, 7, and 8 shows the point of failure. I should like to ask about the shape of the latter part of the curve. Is there a change which can be identified with the start of cracking? This would seem to be an important application of the method, as the start of cracking is fully as significant as the final fracture, but it is difficult to determine experimentally.

MR. P. E. CAVANAGH (*author's closure*).—I should like first to reply to Professor Moore's questions regarding a shortened endurance test with a carefully qualified "maybe yes." I may say that during this work we have naturally had in mind that it might prove to be possible to use this equipment to shorten the time of fatigue tests in spite of all the disappointments met with in the past in attempts to apply somewhat similar methods to this problem.

On the basis of these results, there is a very distinct difference between curves obtained above and below the endurance limit during 100,000 stress cycles, for the metals tested. There is no possibility of obtaining a reasonably accurate value for the endurance limit at present by this method. It can be stated that a given load is above the endurance limit, but the determination of an acceptable value for the endurance limit must be determined by long-time tests. This still leaves the possibility of locating the approximate endurance limit relatively quickly by such a method and only running long-time tests at loads near the endurance limit.

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[5] Research Professor of Engineering Materials, Emeritus, University of Illinois, Urbana, Ill.

[6] Metallurgist, National Bureau of Standards, Washington, D. C.

The results for the non-ferrous metals tested were quite satisfactory. The sensitivity scale is the same for all the curves illustrated. With greater sensitivity, much greater changes may be obtained on the recording meter without exceeding the maximum practical sensitivity of the instrument. Everdur was included in these tests because it gives relatively large changes in core loss curves compared to other non-ferrous materials.

I would emphasize that this work only shows that it is possible to determine that a working part made of the metals tested is being subjected to a load above its endurance limit.

In reply to Mr. Thurston's question concerning the rise in temperature as the speed of testing increases, a rise in temperature will cause the second portion of these curves to become steeper. Differences in plastic strain would cause the curves to change in the opposite direction, in the same manner observed, with increasing speed. There is undoubtedly some temperature effect, but the major effect is from differences in plastic strain.

The reference to other work along the same lines where hardness tests were taken is very interesting. Some of the results obtained in the present work were surprising, to say the least, and are certainly being followed up. Hardness readings taken by Jenkin and Lehmann were made on fractured samples after failure. The hardness tests reported in this paper were made on samples run for only a portion of their endurance life. Why totally different trends were shown is not clear at present.

The Cyclograph has not yet been used to investigate healing by understressing, but possible healing produced by heating and long recovery periods has been investigated and reported in a previous paper.[7]

The transverse crack in the Everdur samples did not start from a point on the seam. Two other samples from the seamed bar also failed in this manner and in each case the transverse crack started about 90 deg. from the seam.

The longitudinal crack developed from the seam extended completely through the sample so that stress conditions were far from normal after this crack developed.

The curves illustrated in Figs. 3, 7, and 8 for magnetic metals are all of the general shape of the curve for S.A.E 1065 tested at 65,000 psi. in Fig. 3. In all samples tested, the first portion of the curve indicates the initial changes in internal stresses. If the stresses exceed a certain limiting value, continuing plastic strain is initiated. The second section of the curve indicates the rate and severity of plastic action leading to failure, before visible cracks are formed.

Most samples of magnetic materials tested have shown a marked change in slope of the curve just before visible cracks appear. From this point on, the curve is usually not the smooth line illustrated, but a regular oscillation about this average line. This behavior is probably due to the periodic building up and relief of internal stresses during propagation of fatigue cracks. The effect cannot be seen at all in some samples, while it is very pronounced in all samples of some steels. The mode of cracking, the difficulty of propagating a crack, and the test load applied will all influence the form of this portion of the curve. From a practical standpoint the second section of these curves is the most interesting.

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[7] P. E. Cavanaugh, "Some Changes in Magnetic Properties of Steels and Wire Rope During Fatigue Failure," Canadian Mining and Metallurgical Bulletin, July, 1947.

A NEW TYPE OF MAGNETIC FLAW DETECTOR[*][1]

BY CARLTON H. HASTINGS[2]

SYNOPSIS

The investigation reported herein is an attempt to develop a practical inspection test method, including instrumentation, for the inspection of the bore of ferrous tubes or cylinders. A review of existing methods[3] which might be applicable to such a problem showed that the development of an entirely new method, combining the advantages of several existing methods, was in order. Consequently, the use of an electromagnetic pickup coil, sensitive to the magnetic leakage caused by flaws in steel magnetized by direct current and connected to an electrical indicator or recorder, was investigated. The work represents an attempt to substitute instrumentation for the magnetic particles of the Magnaflux method. Cracks in the bore surface of tubes, as well as on flat, accessible surfaces, were readily detected and chart records showing their presence were made. Deeper surface cracks caused larger instrument deflections than did the more shallow cracks. By adjusting the sensitivity of the indicating device, the shallow cracks could be passed over and only the larger ones indicated. Surface cracks as shallow as 0.05 in. were detected by increasing the sensitivity of the instrument. A subsurface crack was also successfully located.

Because instrument indicating or recording is used, the inspection process may be speeded up considerably as compared to methods requiring visual examination subsequent to testing. The author considers that this new tool paves the way toward semi-automatic inspection of parts which previously required tedious visual procedures. The possibility of calibrating the instrument to measure surface-flaw depth is also considered. Quantitative data, the use of d-c. magnetization and leakage fields to detect subsurface flaws, and simple instrumentation are available for the first time.

A particularly difficult situation has been encountered in connection with inspection of inaccessible surfaces such as the bore of steel tubing or hollow steel parts. In the latter part of the war a solution to this problem became a crying need because of the necessity for improving the methods for inspecting cannon-tube bores. Visual inspection of the bore surfaces of these tubes was and is still being carried on by means of the optical borescope. Proof tests are conducted as a final acceptance test on such tubes and as a supplement to other inspection tests. Some tubes having satisfactory metallurgical structure were failing during proofing, presumably due to flaws which were missed by the borescope. It is readily understandable that machining operations such as boring or honing might lap over heat-treating cracks, thus making them impossible to detect visually. Also, visual methods, even when ordinarily reliable, are extremely dependent upon the observer, fatigue and carelessness being of prime importance. In short, for applications of the above type, such methods are far too time-consuming and unreliable.

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] The statements or opinions in this article are those of the author and do not necessarily express the views of the Ordnance Department.

[2] Physicist, Watertown Arsenal, Watertown, Mass.

[3] Since the publication of the preprint of this paper a patent has been discovered which outlines the basic idea involved. (U. S. Patent 1,896,737 Method and Apparatus For Testing Rails or the Like, Theodor Zuschlag, February 7, 1933.) It is believed, however, that instrumentation for practical utilization of the idea has never been available commercially.

An investigation of other test methods with regard to their applicability to this problem included consideration of Magnaflux (and Magnaglo), Zyglo (penetrating oil methods), and ultrasonic methods of inspection. Penetrating oil methods (including Zyglo) are not practicable because their success is dependent upon a subsequent visual examination which, in the case of tube bores, requires the use of the borescope. Also, the flaws involved may not be open to the surface, thereby defying detection by this method. Magnaflux and Magnaglo fail to satisfy requirements because they also require the visual study after application. Highly trained personnel are needed to carry out inspection by these methods and little saving in time would result. The recently developed ultrasonic techniques do not offer much hope of a ready answer to the problem because of the unfavorable orientation of the cracks in question, since they usually occur parallel to the axis of the tube and lie in a radial plane. Radiographic methods are also ruled out of the picture because of the unfavorable flaw orientation and because of the shape of the piece to be examined.

A consideration of the advantages and limitations of the above methods of test leads one to conclude that a new method employing d-c. magnetic fields and electrical indicating or recording apparatus would be desirable. As indicated by experience with Magnaflux testing,[3a] d-c. magnetization makes possible the detection of flaws that may be completely covered by sound metal. Flaws in thick sections and located as much as $\frac{1}{2}$ in. below the surface have been detected by this method. The substitution of electrical indicating or recording for the magnetic particles of the Magnaflux method would make remote operation of a detecting device possible. A device might be constructed which would be sensitive to the variations in d-c. magnetic fields at the surface of a piece to be tested, particularly the leakage fields due to flaws such as cracks, and still be of a small enough size to be inserted in tube bores or other critical but hard-to-get-at spots. Detecting coils have been available for some time for the exploration of the exterior surfaces of ferrous tubes, but in order to obtain inductive pickup, a-c. magnetic or electric fields have been employed.[4, 5] These methods are, therefore, limited to the detection of flaws that are on or relatively near the surface because of the shielding "skin effect" which is present when a-c. fields are employed even in nonmagnetic materials. They also require that the motion of the tubing through the detecting coils be maintained at a uniform rate, thus involving more cumbersome equipment. More recently a method for bore testing has been developed which uses direct current for magnetization of the part to be inspected, but requires that the detecting coil be rotated at a constant speed to obtain uniform inductive pickup.[6] This method involves a motor, gear train, shaft, and bearings, etc.

[3a] Carlton H. Hastings, "The Magnetic Powder Method of Inspecting Weldments and Castings for Subsurface Defects," *The Welding Journal*, Welding Research Council Supplement, January, 1943.

[4] Theodor Zuschlag, "Magnetic Analysis Applied To The Inspection of Bar Stock and Pipe," ASTM BULLETIN No. 99, August, 1939, p. 35.

[5] H. C. Knerr, "Electrical Detection of Flaws in Metal," *Metals and Alloys*, Vol. 12, October, 1940, p. 464.

[6] John R. Drummond and Martin Fleischmann, Defect Detector For Tubes, U. S. Patent 2,308,159.

AN APROACH TO THE PROBLEM

In searching for a method which might overcome the above disadvantages, an old magnetic principle was called to mind. This principle is new to nondestructive testing but is common in other electrical circuits. The power-supply filter choke may be taken as an everyday example. In such applications one is concerned with the behavior of an iron-cored coil when it is under the influence of combined a-c. and d-c. magnetic fields. Ferromagnetic materials do not have a constant permeability. Instead, this characteristic changes with the degree of magnetization. If an iron-cored coil is subjected to both a-c. and d-c. magnetic fields, the permeability of the core material and the inductance of the coil will change if the magnitude of either field is varied. Since our problem is to detect variations in d-c. magnetic fields, it was considered that the inductance of a coil through which alternating current is passed might vary when it encountered local variations (leakage fields caused by flaws) in a d-c. field at the surface of magnetized parts undergoing test. In such a procedure, the detecting instrument operates on alternating current and the part to be inspected is intentionally magnetized *only with direct current.*

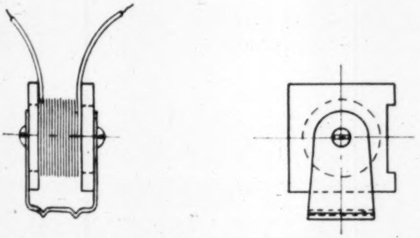

FIG. 1.—Sketch of Original Coil and Shunt, 850 Turns of No. 36 S.C.C. Wire.

FUNDAMENTAL INSTRUMENTATION

In order to test this principle as applied to flaw detection, a coil was constructed on a small iron form and shunted by a thin strip of a nickel-iron alloy (see Fig. 1). This alloy is particularly sensitive to variations in weak magnetic fields and does not remain magnetized when the magnetizing force is removed. In order to obtain high sensitivity in detecting changes of the coil inductance, it was connected into an electrical bridge circuit. This type of circuit makes possible an electrical balance between the detector coil and another identical coil. When the magnetic characteristics of one coil are altered and the other coil is unchanged magnetically, the system becomes unbalanced. The amount of unbalance (appearing as a current or voltage change) may be indicated by a meter, recorder, or other indicator connected in an appropriate manner. An amplifier connected between the bridge and the indicating meter may be used to magnify the unbalance by a factor of 10,000 times or more if desired. Thus it can be seen that a change in the detector-coil characteristics of only a very slight amount may cause a relatively large deflection of the indicating meter.

Test Specimens:

The first test specimen provided a very easy case of a simulated flaw. Two steel plates were butted together edgewise and welded across the ends to give electrical continuity. A direct current of the order of 200 to 400 amp. was passed through the plates parallel to the separation between them. This high amperage current, obtained from a heavy-duty copper oxide rectifier, was brought into the plates by contacts clamped thereto. The search coil, connected to the bridge by means of a flexible cable, was placed on one of the abutting plates away from the separation and the bridge adjusted to balance. By moving the detector coil over the gap, a deflection of the meter was noted. It was not necessary to have the coil in motion to get a deflection of the meter, a fact peculiar to this method of detection. If the coil was placed over the gap, the meter remained in its deflected position until the coil was again moved away from the gap. It was then known that a search coil of this type would respond to crack-like voids. The question remained, "Will it detect a true crack of the size encountered in practice?"

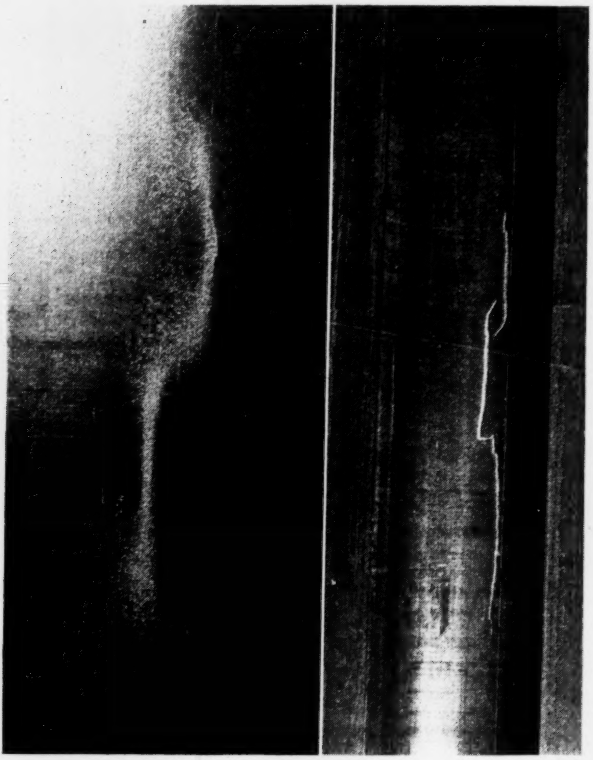
Exterior Bore Surface

Fig. 2.—Magnetic Particle Pattern Caused by Crack in Cylinder.

A second test specimen consisted of a block of 3-in. thick steel having a quench crack system on one of its larger faces. By grinding off successive thin layers of this face of the block and testing between grinding operations, a point was finally reached where the crack caused only a barely perceptible meter deflection. A measurement of the remnants of the crack indicated a depth of about 0.05 in. Thus it was considered that the principle was capable of yielding adequate sensitivity.

Still another test was made on a hollow cylinder about 20 in. long with a $1\frac{1}{4}$-in. wall thickness. This cylinder had previously been submitted to a hydrostatic pressure test and had failed by cracking. The resulting crack was visible for about $\frac{1}{2}$ in. of length on the outside surface. By sectioning longitutudinally after completion of tests, it was found that this crack was about 5 in. long on the inside surface. The crack may be visualized as having a roughly triangular shape as viewed on a section through the wall, the triangle having its base on the inside surface and its apex at the exterior surface. For tests conducted on this cylinder, a cable threaded through its bore and attached to a power unit provided approximately 1600 amp. turns d-c. for magnetization. While maintaining this magnetizing force, some dry Magnaflux powder was dusted onto the exterior surface and a powder pattern typical of this type of flaw was formed (see Fig. 2). That is, the pattern was narrow and sharply defined at the point where the crack came to the surface and broadened out to a wider pattern fading off at both ends of the visible crack. To those familiar with such powder patterns this indicates a variation in the distance of the crack from the surface. At the narrowest part of the pattern a surface flaw was indicated; at wider parts a subsurface flaw was indicated. The total length of the powder pattern was $4\frac{1}{2}$ in. Exploration of the exterior surface by means of the magnetic detector coil also indicated the presence of the crack throughout a length of $4\frac{1}{2}$ in. The magnitude of meter deflection was greatest at the point where the flaw came to the surface and decreased on either side of this location. In this particular instance, at least, results obtained with the magnetic detector coil compared favorably with those obtained from the Magnaflux test on both the surface and subsurface portions of the crack.

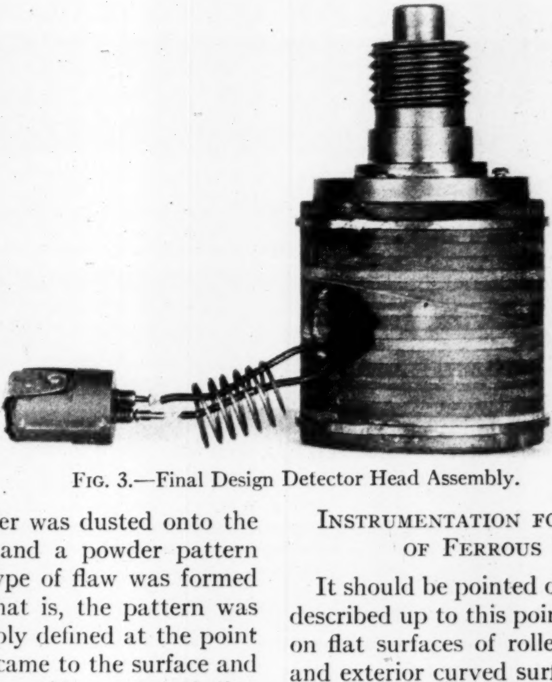

FIG. 3.—Final Design Detector Head Assembly.

INSTRUMENTATION FOR INSPECTION OF FERROUS TUBES

It should be pointed out that the work described up to this point was performed on flat surfaces of rolled plate material and exterior curved surfaces, some work not described being done on the as-cast surface of cast steel plate. Thus, application to such inspection is indicated. The design of instrumentation for the application of the method of test to production inspection of the bore of tubular parts was next to be considered. This problem was resolved into two parts, the development of an adaptor to hold the detector coil properly in the tube bore for scanning, and the development of suitable electrical apparatus to indicate or record the desired information obtained during tests.

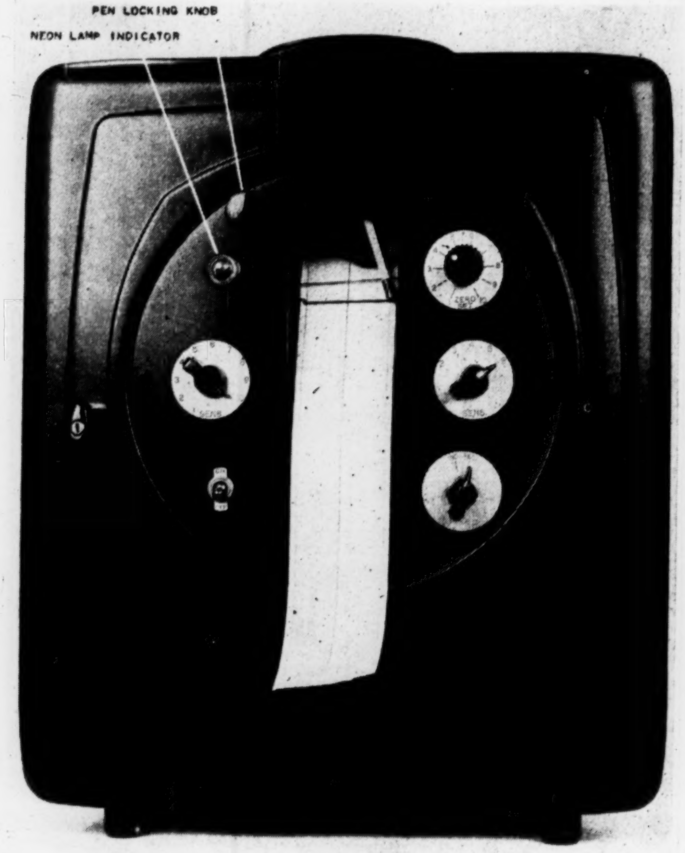

Fig. 4.—Front Panel of Recorder-Indicator.

Design for Detector Head:

The principal function of the detector head is to prevent the coil from tilting or turning in the tube and thus altering its contact with the surface of the metal. After trying several schemes, a design which seemed satisfactory for this purpose was evolved (see Fig. 3). Two copper wires were wrapped around a cylindrically shaped piece of laminated plywood to serve as bearing bands. They provide a reasonably good fit without wear to the surface being examined and decrease sliding friction. A hole was bored in the side of the piston-like head to accommodate a helical spring and the detector coil. The spring, pushing on the coil, provided uniform contact pressure with a bore surface under examination. Another hole was bored from one end of the head to the other to accommodate the electrical leads to the coil. For these test models of the device, a long tubular handle was fastened to the back of the head by which the assembly could be manipulated through the bore. The electrical leads were brought out through this handle to the rest of the apparatus.

Development of Electrical Apparatus:

The electrical components, apart from the detector head, may take many forms. The particular unit chosen was available from a commercial instrument maker[7] and embodied several features which are desirable for production testing. A picture of this apparatus is shown in Fig. 4. Simpler instrumentation is possible with some sacrifice in versatility. By reference to the above figure one can note a narrow strip chart upon which a pen-and-ink chart record of variations in the bridge output voltage can be made. Bridge components and amplifier circuits are contained in this unit. Also, by operating a switch, a neon flasher lamp may be substituted in place of the recorder. Although the advantages of a permanent chart record of inspection operations will be obvious to the reader, it must be stated that the rate of inspection which can be followed by an electromechanical recording pen is definitely limited. Therefore, for inspection of a more rapid and cursory nature, the inertia-less neon lamp offers advantages. The sensitivity of response of both pen and indicating lamp is adjustable over wide limits. The complete inspection unit as set up in the laboratory is shown in Fig. 5. The small box to the left of the recorder is for matching the electrical characteristics of the detector and balancing coils before a test is made. Instrumentation can be further simplified by elimination of the balancing unit with some sacrifice of sensitivity.

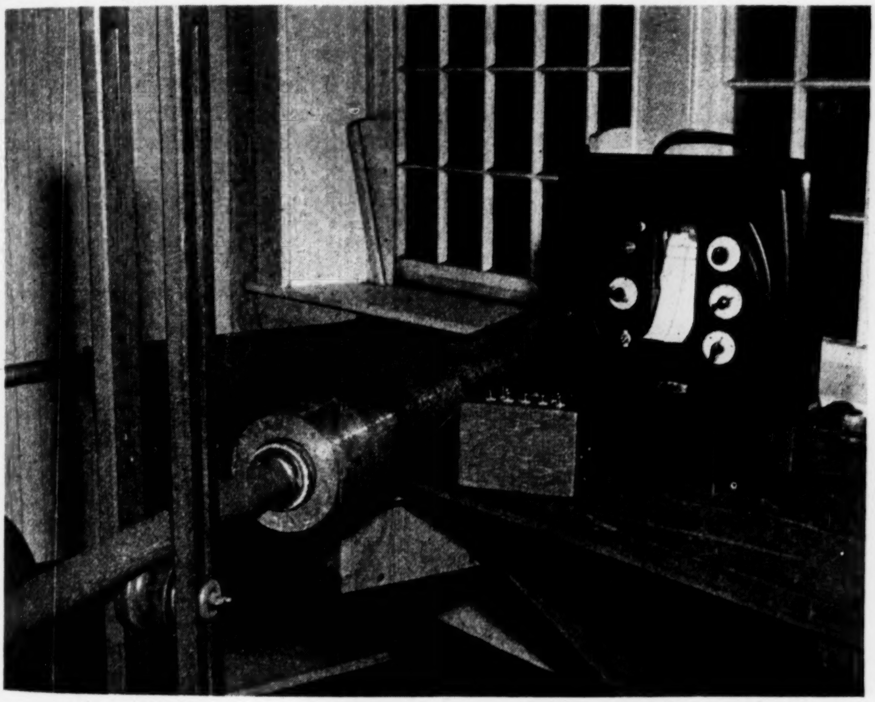

FIG. 5.—Complete Inspection Setup in Laboratory.

Tests on Thick-Walled Tube:

The tests on a length of thick-walled tube are of practical interest. This particular tube had been rejected during manufacture, on the basis of borescope examination, the report stating that the nature of the bore-surface flaws causing rejection was not definitely known. Borescope examination of the full-length tube by the author indicated two possible surface cracks, but because of the scratched and pitted surface a definite statement that cracks existed could not be made. The bore had been subjected to a honing operation, and the resulting mirror-like finish (except where scratched and pitted subsequent to machining) caused highlights which made visual examination difficult. A 4-ft. length of this tube containing the suspected flaws was removed for further study. Since a magnetizing cable could not be located in the bore at the same time that this surface was being scanned, only two other possible magnetizing methods remained: one of these was to pass the magnetizing current through the metal of the tube; the other method being to magnetize by means of the cable threaded through the bore, remove the cable, and rely upon the residual magnetism retained by the steel. The residual method was tried and found to be adequate for the detection of existing surface flaws. Preliminary examination of this tube showed more than two indications of the type previously associated with crack-like flaws. Other recorder deflections seemed to be coincident with rusted areas. A rough machine-cut through the bore eliminated the possibility of interference from rusted areas, and it was thereby shown that the presence of red rust will cause confusion when using the instrument. However, it is believed that most applications of the flaw detector can be made on a scaled or freshly machined surface or at least on surfaces not covered with red rust. Retesting the clean bore surface of the tube showed four distinct crack-like deflections which appeared as a rapid swing of the recording pen followed by an equally sharp pen reversal as movement of the detector coil was continued beyond the crack. A re-check of the bore surface with the borescope showed four unmistakable cracks. The breaking of the metal chip during machining left a shiny edge on one side of each crack which delineated each separation. It should be pointed out that four crack-like indications were obtained when testing the original honed surface with the magnetic detector whereas two of these cracks could only be suspected on borescope examination. Another important point could be made of the fact that the flaws were all surface flaws, giving the borescope an advantage. If the cracks had been lapped over or subsurface, the borescope would have missed them entirely; but, based on the previously cited experiment with subsurface cracks in the hydrostatically ruptured cylinder, the magnetic detector would still have been able to pick them up. Closer examination of this tube showed that the length of the cracks could be determined easily and accurately by proper scanning. Typical chart records of cracks in this tube are shown in Fig. 6, in which (*b*) illustrates the use of a higher sensitivity than (*a*), thus the greater deflection. Two cracks are recorded in (*a*), only one in (*b*). The magnitude of deflections indicates relative depth of cracks.

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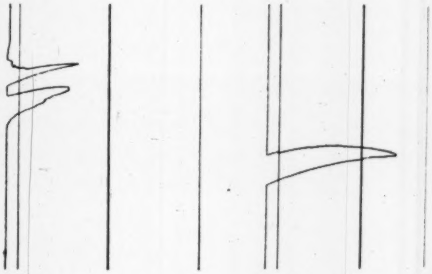
[7] Foxboro Co., Foxboro, Mass.



(*a*) (*b*)

FIG. 6.—Typical Chart Records.

APPLICATION TO PRODUCTION INSPECTION

The consideration of procedures for application of the magnetic detector to shop inspection of heavy walled tubes has brought forth the following ideas. The tubes may be magnetized by means of a direct current passed through a cable threaded through the bore of the tube. The residual method is adequate for the detection of near surface flaws. An automatic mechanism can be arranged to pull the detector head through the bore after this cable has been removed. If the tube is mounted on end rollers and turned slowly as the detector is being drawn through the bore, the head will scan a spiral path over this surface. By adjusting the rate of turn and forward motion to suit the size of the detecting coil, the entire surface of the bore can be examined with one pass. Further, if the pulling mechanism is synchronized with the chart drive of the recorder, it would be a simple matter to determine the exact location and extent of bore cracks by an examination of chart records. The addition of a linear and circular scale associated with the cable or rod used to pull the head would allow the operator to determine the longitudinal and o'clock position of flaws when the neon indicating lamp is used in place of the recorder.

SUMMARY

A new method of detecting flaws in ferromagnetic materials has been developed which combines the variable sensitivity of electronic indicating or recording with the possibility of detecting relatively deep-seated flaws. This possibility has not heretofore been realized by methods depending upon a-c. magnetic fields for detection. The sensitivity attainable may be expressed in terms of the depth of the shallowest surface flaw detectable. This figure is at least as small as 0.05 in. The extent to which subsurface flaws can be detected has not been fully determined. Because of the small size of the search unit, it is applicable to the bore of hollow parts which are relatively inaccessible. The method described is not suited to the examination of rusted surfaces but is not bothered by scale or rough machined finishes. Finishes as rough as 500 microinches rms. can readily be inspected. Also, an unexplored possibility is opened up by the method. Since quantitative data are obtainable in the form of a chart record or meter readings, it may be possible to correlate the magnitude of deflections with the depth of detected flaws. In other words, it may be possible to measure hidden cracks as well as locate them if a number of parts of the same type are to be examined. Further work on the subject is expected to clarify this possibility.

DISCUSSION

Mr. D. M. McCutcheon.[1]—The barrel must have been magnetized prior to using the test probe. Is that correct?

Mr. C. H. Hastings (*author*).—That is correct. A central conductor with current to give 1600 ampere turns was used for magnetization. The conductor was then withdrawn and the residual magnetism utilized for testing. The barrel was not of a particularly highly retentive alloy. The success obtained in this test indicates that for the detection of surface flaws, relatively weak magnetic fields are adequate.

If it is desired to detect subsurface flaws, however, it may be necessary to use continuous magnetization.

With regard to the chart record of results, I should like to explain a little trick which produced a more easily readable record. If the pen is zeroed on the center of the chart it can swing either way with equal sensitivity. Pen fluctuations may be caused by such things as hard spots, segregations, stress concentrations, and other variables which may be present in the metal under test. These pen fluctuations confuse the chart record and are usually of no interest when looking for cracks. To simplify chart reading, the pen was zeroed slightly off scale so that the lesser background fluctuations did not record. This explains the nice straight line on the edge of the chart. Since the pen will always swing the same way for a crack, these flaws will always give rise to a peak in the trace if the magnetic disturbance due to the flaw is greater than the magnetic disturbance due to the other nonrelevant variables. However, if the flaws are smaller than the surface discontinuities or other variables, difficulty will be experienced in their detection.

Mr. McCutcheon.—This could also have been accomplished in your electrical circuit.

Mr. Hastings.—Yes. I had thought during the development work that one of the nice circuit details to be worked out in a later model would allow indication of sharp discontinuities such as cracks but which would not allow indication of such things as segregation or variations in hardness or stress covering a wider area. Cracks or other sharp discontinuities give rise to a steep peak on the curve because of the relatively short-time constant of the electrical signal resulting. A circuit could very likely be worked out which would discriminate in favor of these short-time constant signals caused by cracks, thereby eliminating the background hash automatically.

Mr. McCutcheon.—Did you find it necessary to use a-c. current in the coil? Simply moving a coil past the discontinuities would create a voltage peak as a result of the magnetic disturbances.

Mr. Hastings.—The method suggested is used in a number of existing test methods and gives rise to one of the bad features of such instrumentation. In such cases one is generating an emf. in the coil by the relative motion between it and the d-c. leakage field. A uniform rate of coil movement must be maintained in order to obtain emf.'s which can be related in magnitude to the flaws. The faster you scan, the greater the emf. and pen deflection. When using the method described in this paper, the emf. is independent of the speed of scanning, and the speed of response of the indicator is the limiting factor on how fast you can go. If the coil is halted over the flaw, the pen will remain deflected. Its deflection does not require coil movement. It is necessary to use alternating current in the coil and bridge to allow measurement of the search coil inductance. A variation in this parameter, caused by the flaws in question, is the quantity to be observed.

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[1] Chemical and Metallurgical Research, Applied Physics Div., Ford Motor Co., Dearborn, Mich.

MR. S. A. BROSKY.[2]—Did I understand the author to say that the smallest depth that could be detected was fifty thousandths?

MR. HASTINGS.—The smallest as yet is 0.05 in.

MR. BROSKY.—That does not seem to agree with the chart and your background flaws. That is a pretty rough cut in the bore of the barrel, because the background was rather smooth and the defect to be detected must have been of sizable depth.

MR. HASTINGS.—The flaws indicated by the chart records were of quite sizable depth and the background was suppressed by the method of zeroing described previously. However the evaluation of sensitivity was not made on this gun tube; it was made on a flat block having a Blanchard-ground finish. The grinding marks might have had something to do with the limit of 0.05 in. That is why I stated that I think we can perhaps do better than 0.05 in.

MR. BROSKY.—Would you, if you got no indication, accept that gun barrel to be satisfactory for service?

MR. HASTINGS.—I would say that it had no flaws greater than a certain depth say 0.05 in.

MR. BROSKY.—I cannot see how that could be. I have seen 8-in. marine shafts fail from a prick punch mark on the surface, and I have seen other steamboat shafts fail from a tack-weld mark.

MR. HASTINGS.—I did not mean to imply that I would accept or reject a gun barrel with flaws 0.05 in. in depth. I stated that I would agree that there were no flaws of that size present if not shown by the detector. As is the case with any inspection method, the setting up of standards of acceptance is entirely apart from the location of possible flaws. Acceptance on the basis of what one finds is subsequent to the actual testing. Unfortunately any testing method may appear inadequate if used in connection with improper acceptance limits. By using poorly established standards, a piece accepted as satisfactory may fail in service, reflecting upon the test method. Also unnecessary rejections may be made.

MR. BROSKY.—I am wondering whether we should throw out the magnaflux testing equipment.

MR. HASTINGS.—I would say absolutely not. On what basis would you do so?

MR. BROSKY.—I believe that magnaflux will give you more sensitivity in locating surface flaws which really have everything to do with the acceptance of the gun barrel.

MR. HASTINGS.—Magnaflux has many useful applications. In the case of gun tube bores and similar applications, it is not first a question of sensitivity but one of practicability at this time. Magnaflux is not a practicable inspection method for this problem. That is why we developed the new method. Even if you can apply magnaflux, it can be done with great difficulty, you still cannot see the results of the test without a boroscope. The magnetic particle patterns may be six or more feet from the end of the bore and quite inaccessible for examination without some optical aid. We have tried the boroscope and were not able to distinguish between a magnetic particle pattern and a piece of lint from the rag used to clean the bore. Boroscope examination and retesting are so tedious as to make the whole method prohibitive for production testing.

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[2] Department Head of Radiography, Pittsburgh Testing Laboratory, Pittsburgh, Pa.

MR. BROSKY.—Well, that sensitivity limit of fifty thousandths worries me.

MR. HASTINGS.—Perhaps, but you must admit it is an improvement when considering the practicability of available methods for this problem. This sensitivity limit might be improved by further development.

MR. L. S. BIRKS.[3]—Does this method detect things other than cracks, such as a change in the grain size which might affect the magnetic characteristics? How would that appear in the record?

MR. HASTINGS.—If the variation in grain size influences the magnetic character of the metal under test it will probably cause instrument response along with the other so-called background variables. However, since such a variation would probably cover some area of the surface its effect on the results would not be readily confused with that caused by a sharp discontinuity such as a crack. The indication would be broader or less sharply peaked than that of a crack. Therefore, either by experience in examining chart records or by proper design of circuits to discriminate against long-time constant signals, this problem can be largely eliminated.

Such variables do enter the picture, as is the case with all magnetic methods that I know of, however, and must be dealt with in some way.

MR. MCCUTCHEON.—What influence does varying the frequency of the current in the search coil have on its sensitivity?

MR. HASTINGS.—I have recently made some tests regarding that point. Varying the frequency of the bridge or measuring circuit has a bearing on the over-all sensitivity of the method. Smaller variations in search coil inductance can be detected by the bridge if it is operated at low frequencies of the order of 60 cycles. Some tests were made on a search coil using a General Radio impedance bridge excited at various frequencies from fifty cycles to one megacycle. One measurement of the coil inductance was made with its core in place and another with the core removed. The greatest difference in readings was noticed at the lowest frequencies.

MR. J. S. WORTH.[4]—What type of indication is produced by nonmetallic inclusions?

MR. HASTINGS.—This has not been investigated. It is one of the studies which should be made, however. Regarding flaws other than cracks, magnetic particle tests have been made on a large tubular forging furnished to us for study by the Bethlehem Steel Co. and have shown the presence of flakes in the bore of this piece. Having no adapter large enough to fit the bore of this forging the search coil was manipulated by hand. Thus my tests were of limited scope. However, by passing the coil back and forth across a flake I was able to get a definite response. Residual magnetism and high instrument sensitivity were employed. It is possible that if I had surveyed the entire bore surface, background variables might appear as gross as the flakes at that high sensitivity level, thus making it difficult to distinguish the flaws. I hope to make a complete survey of this tube in the future.

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[3] Naval Research Laboratory, Navy Department, Washington, D. C.

[4] Assistant Metallurgical Engineer, Bethlehem Steel Co., Bethlehem, Pa.

MR. GEORGE KEINATH (*by letter*).[5]—The author has already mentioned that the advantage of a permanent chart record of the inspection is linked to the serious disadvantage of slow operation, caused by the excessive mechanical lag of the pen mechanism of conventional recording systems used for the test.

No time limits for the test are given, but we can make a guess:

1. We can assume that we need a current impulse of at least 0.1 sec. duration to get a good deflection of the pen.
2. The smaller the detector head area, the better for the detection of flaws. A large head will integrate over a large area and the same crack will give a much smaller percentual current change than a very small detector head would give.
3. Assuming 3 mm. crack width and a time of 0.1 sec. for the duration of the current impulse and an inner circumference of the bore of 300 mm. (100 mm. diameter), the time for one revolution of the tube under test cannot be less than 10 sec.
4. For a tube length of 3000 mm. we would need 1000 revolutions of the tube under test (or the detector head) to scan the whole length every 3 mm.

With more modern electrical recorders than the one used by the author, it is possible to get much higher speed, as high as mechanically possible for the rotation of tube or detector head and at the same time we can get a far more perfect recording, giving intensity of the impulse caused by the crack, longitudinal as well as o'clock position of the crack, all at the same time, in one picture of the bore surface and the flaws in it.

We use a facsimile recorder with volt-sensitive paper of the same type as used for transmitting telegrams and newsprint. The drum is rotated in synchronism with the tube (up to 5 revolutions per second) and the stylus is connected in the same way as the pen of the present ink recorder. The stylus is travelling along the drum in synchronism with the travel of the pickup relative to the tube.

With no flaw, light continuous markings are made with 1000 cycle discharges, giving a uniform gray pattern. As soon as the pickup finds a flaw, light or deep, it generates a proportionally higher diagonal voltage in the a-c. bridge and makes a heavier mark whenever the pickup comes over the flaw area, thus giving a perfect picture of the pattern, the shape and location of the flaw on the chart.

Recording is all-electronic. There is no mechanical inertia and the duration of an impulse may be as short as 0.0001 sec. to give a full recording.

With only one revolution per second, the speed of the new electronic "Sweep Balance" recorder is already at least ten times higher than with the present recorder. The testing time, to use the figures of the above example, is reduced from about 3 hr. to less than 20 min., and at the same time a smaller detector head could be used.

Instead of a drum recorder, a strip chart recorder with rotary helix can also be used with a maximum normal speed of 6 recordings (six revolutions of tube or pickup) per second, if this is mechanically possible.

MR. HASTINGS.—In spite of the fact that Mr. Keinath's assumptions lead to an erroneous impression of the time necessary to examine the tube in his example with the instrumentation employed by me, any improvement in the speed of response of the apparatus associated with the search coil would be considered a step in the right direction. To elaborate on the above, a 3-mm. (about $\frac{1}{8}$ in.) wide crack would be much wider than average. Also, an infinitely small size of detector head sensitive surface has been assumed which offers practical difficulties. Assuming a 0.3-mm. width for a longitudinal crack and a shunt strip 12.0 mm. wide, it would take approximately 2.5 sec. to scan the 300 mm. circumference of the bore of a tube if a current pulse duration of 0.1 sec. is required to operate the instrument. The current pulse would persist for the length of time necessary for the full width of the sensitive coil to pass across the crack. For a tube length of 3000 mm. and a shunt strip 12.0 mm. long it would take about 250 revolutions to scan this length every 12.0 mm. necessary to cover the surface completely. These figures give a total time of examination of about 10 min. rather than 3 hr. for a case using more practical figures.

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[5] Consulting Engineer, Larchmont, N. Y.

Nevertheless an instrument requiring pulses of only 0.0001 sec. duration would do the same job in a small fraction of the above time and would permit use of the smallest practicable search unit to gain greater discrimination as pointed out by Mr. Keinath. Thus the relatively inertialess recorder using volt-sensitive paper and recording position as well as magnitude of detector signal would seem to offer interesting possibilities for improved performance.

FATIGUE CHARACTERISTICS OF ROTATING-BEAM *VERSUS* RECTANGULAR CANTILEVER SPECIMENS OF STEEL AND ALUMINUM ALLOYS*

By F. B. Fuller[1] and T. T. Oberg[1]

Synopsis

Fatigue tests were conducted in Krouse vibratory nonrotating cantilever beam (fixed deflection) and R. R. Moore rotating simple beam (constant load) fatigue testing machines to compare the fatigue characteristics in reversed bending for material tested in these machines in the form of round rotating simple beam and of rectangular cantilever beam specimens. Specimens were machined from an extruded shape of 75S-T, 24S-T, 14S-T, and R303 aluminum alloys and from chromium-molybdenum S.A.E. 4130 flat steel plate of several degrees of hardness. The fatigue characteristics in general for the material in the form of rectangular specimens were consistently lower than those for the material in the form of round specimens.

Two bending fatigue testing machines used extensively for determining fatigue strength of materials are the R. R. Moore and the Krouse plate types. From tests in these machines there is a considerable amount of fatigue data available for round rotating simple beam specimens and vibratory cantilever (nonrotating) beam specimens. However, published data for direct comparison of the fatigue characteristics of materials tested by these two methods are very limited. All data indicate that the properties are higher for the round specimens (**1, 2, 3, 4**).[2] For purposes of obtaining comparative data on these two machines for the same material and using recognized types of test specimens, an investigation was made on material from a steel plate. Data obtained previously from several aluminum alloys from the same type of extrusion are also included. It is believed that, in general, the history of each specific material has been eliminated as a factor in comparison. The factor of shape effect was not considered in analyzing the data.

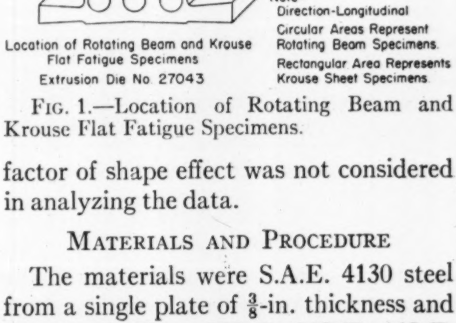
Flat Fatigue Specimens
Extrusion Die No. 27045
Note -
Direction-Longitudinal
Circular Areas Represent Rotating Beam Specimens.
Rectangular Area Represents Krouse Sheet Specimens.
Location of Rotating Beam and Krouse Flat Fatigue Specimens
Extrusion Die No. 27043

Fig. 1.—Location of Rotating Beam and Krouse Flat Fatigue Specimens.

Materials and Procedure

The materials were S.A.E. 4130 steel from a single plate of $\frac{3}{8}$-in. thickness and aluminum alloys 75S-T, 24S-T, 14S-T,

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Chief, and Materials Engineer, respectively, Structural and Mechanical Test Branch, Materials Laboratory, Engineering Division, Air Matériel Command, Wright Field, Dayton, Ohio.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 672.

and R303-T from an extruded shape. The extrusion and the location of specimens therein are shown in Fig. 1.

The length of the static round tension, round rotating beam, and rectangular cantilever beam fatigue specimens from the S.A.E. 4130 steel plate was parallel to the direction of rolling. The types of specimens, Fig. 2, were machined with the minimum diameter of the round specimens and the gage thickness of the rectangular specimens the same and with the axis of the specimens coincident with the axis at mid-depth of the plate. Specimens were rough-machined within 0.030 in. of the finished dimensions and then heat treated as follows: oil-quenched from 1625 F. and tempered to Rockwell C 26 (group 1), to C 34, (group 2), and to C 44, (group 3). The round rotating-beam fatigue and tension specimens in groups 1 and 2 were then machined, turned to the finished dimensions, and those in group 3 were finished by grinding. The gage section of the round specimens was polished longitudinally in a superfinishing machine similar in design to that used by Chrysler Motor Corp. to an average finish of 10 microinches. The flat surfaces were ground after heat treatment and then hand-polished parallel to the length with Nos. 1, 0, and 000 emery paper to an average finish of 20 microinches. The sharp edges in the gage section of the specimen were broken with emery paper. Surface finish measurements were made with a Brush SA-2 Surface Analyzer.

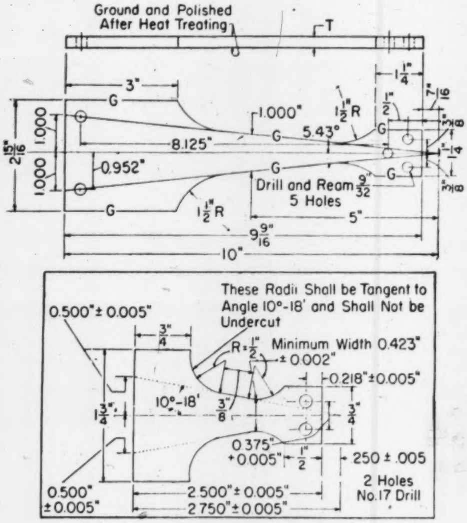

Ground and Polished
After Heat Treating
T
3"
G
1.000"
1½" R
5.43°
1¼"
½"
7/16"
3/8"
¼"
3/8"
8.125"
0.952"
1.000
1.000
2 15/16"
Drill and Ream 9/32"
5 Holes
1½" R
5"
9 9/16"
10"
These Radii Shall be Tangent to
Angle 10°-18' and Shall Not be
Undercut
0.500" ± 0.005"
¾"
R = ½"
Minimum Width 0.423"
± 0.002"
0.218" ± 0.005"
10°-18'
3/8"
¾"
1¾"
0.375"
+0.005"
½"
250 ± .005
2 Holes
No. 17 Drill
0.500"
± 0.005"
2.500" ± 0.005"
2.750" ± 0.005"

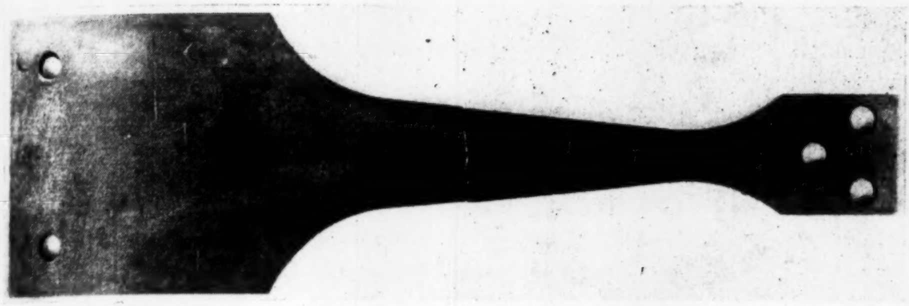
FIG. 2.—Dimensions of Retangular Specimens.

The specimens from aluminum alloy extrusions were located at random throughout the length of the extrusion and were machined and finished similar to those for the steel specimens. However, no surface measurements were made, but it is believed that they would be of about the same order as in the case of the steel. All specimens were coated with a noncorrosive oil after polishing.

The loads required to develop the desired stresses in gage sections of the round and rectangular specimens were calculated by the standard $\frac{Mc}{I}$ formula. In setting up the Krouse machine, the calculated load was applied at the pin connection of the drive mechanism by dead weights and the deflection resulting from this load was measured. The load was then removed, and the drive mechanism was adjusted to give the same

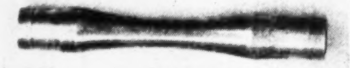
TABLE I.—SPECIFICATION CHEMICAL COMPOSITION, PER CENT.

	Material	C	Mn	P	S	Cr	Ni	Mo	Cu	Si	Mg	Zn	Al
No. 1	Al Alloy 75S-T	...	0.2	...	...	0.3	...	...	1.6	...	2.5	5.6	Rem.
No. 2	Al Alloy 24S-T	...	0.6	...	...	...	...	...	4.5	...	1.5	...	Rem.
No. 3	Al Alloy 14S-T	...	0.8	...	...	...	...	...	4.4	0.8	0.6	...	Rem.
No. 4	Al Alloy R303-T	...	0.3	...	...	0.3	0.1	...	1.0	0.2	2.5	6.5	Rem.
No. 5	S.A.E. 4130 Steel	0.27	0.40	0.04	0.05	0.80	...	0.15	...	...	...	...	...
		0.30	0.60	...	...	1.10	...	0.30	...	...	...	...	...

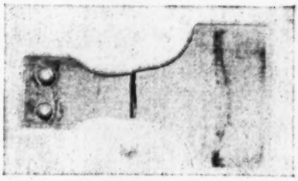


Cantilever flat plate steel specimen, typical fatigue failure.

Rotating beam round steel specimen.



Rotating beam round aluminum alloy specimen.



Cantilever flat plate aluminum alloy specimen, typical fatigue failure.

FIG. 3.—Types of Specimens Used.

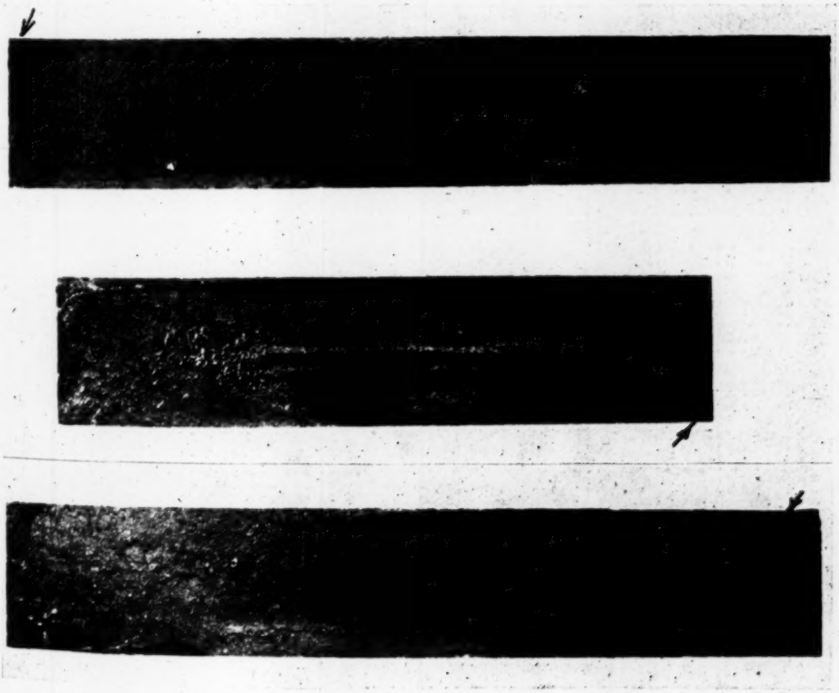


FIG. 4.—Fatigue Fractures in Cantilever Steel Specimens. Arrows indicate the approximate location where the fracture started.

deflection. After the tests were started no further adjustments of deflection were made. The speeds of the R. R. Moore rotating-beam and of the Krouse Machine were 10,600 and 1750 rpm., respectively. The specified chemical composition for the materials are given in Table I. The cantilever specimens (Fig. 2) are the type recommended by the Krouse Testing Machine Co.

TEST DATA AND DISCUSSION OF RESULTS

Typical fatigue failures in the rectangular cantilever specimen are shown in Fig. 3 and a more minute study for the steel in Fig. 4.

Tension and fatigue data are given in Table II.

It is interesting to note that the ratios of fatigue strength (approximate endurance limit) for rectangular to round rotating-beam specimens for aluminum alloys varied from 0.93 to 0.97 and those for the steel specimens varied from 0.85 to 0.64, depending on the hardness. In practically all the tests, sufficient data were obtained to study the fatigue characteristics throughout a fairly representative number of cycles.

The authors used the steel data, Fig. 5, as a basis for their paper, since the round and rectangular specimens were practically of the same depth and prepared so that the surface fibers were approximately the same distance from the mid-depth of the plate—thus the properties of the material in the specimens were the same. The number of round specimens was satisfactory to establish a fairly complete *S-N* curve. However, the number of rectangular specimens was few, consequently resulting in rather in-

TABLE II.—MECHANICAL PROPERTIES OF FERROUS AND NON-FERROUS ALLOYS.

	Material	Tensile Properties			Fatigue Strength, psi. at			Ratio Fatigue Strength to Tensile Strength[a]	Ratio Fatigue Strength Rectangular to Round Specimen[a]
		Tensile Strength, psi.[b,c]	Yield Strength 0.2 per cent offset, psi.	Elongation over 4D, per cent	20 × 10⁶ Cycles	100 × 10⁶ Cycles	500 × 10⁶ Cycles		
ROUND ROTATING SIMPLE BEAM SPECIMENS—0.300-IN. DIAMETER									
No. 1-1	Al Alloy 75S-T	87 600	77 700	14.0	23 000	21 500	21 000	0.245	...
No. 2-1	Al Alloy 24S-T	84 400	65 400	19.0	27 500	24 000	22 500	0.284	...
No. 3-1	Al Alloy 14S-T	75 900	67 900	15.5	25 500	20 500	...	0.270	...
No. 4-1	Al Alloy R303-T	81 600	76 600	14.5	22 500	21 500	...	...	...
CANTILEVER SHEET SPECIMENS—0.093-IN. THICK (RECTANGULAR)									
No. 1-2	Al Alloy 75S-T	...	...	...	23 000	20 000	...	0.228	0.930
No. 2-2	Al Alloy 24S-T	...	...	...	24 000	22 500	...	0.266	0.937
No. 3-2	Al Alloy 14S-T	...	...	...	22 000	20 000	...	0.264	0.976
No. 4-2	Al Alloy R303-T	...	...	...	24 000	...	...	...	...
ROUND ROTATING SIMPLE BEAM SPECIMENS—0.190-IN. DIAMETER									
No. 5-1	S.A.E. 4130 Steel[d]	129 000	118 000	17.0	73 000	...	...	0.566	...
No. 5-2	S.A.E. 4130 Steel[e]	150 000	143 000	14.5	85 000	...	...	0.567	...
No. 5-3	S.A.E. 4130 Steel[f]	206 000	194 000	10.0	115 000	...	...	0.558	...
CANTILEVER PLATE SPECIMENS—0.187-IN. THICK (RECTANGULAR)									
No. 5-1	S.A.E. 4130 Steel[d]	...	...	...	62 000	...	...	0.481	0.85
No. 5-2	S.A.E. 4130 Steel[e]	...	...	...	72 000	...	...	0.480	0.847
No. 5-3	S.A.E. 4130 Steel[f]	...	...	...	74 000	...	...	0.359	0.643

[a] Ratio based on fatigue strength at 100 000 000 cycles for aluminum alloys.
[b] Tension specimen for Aluminum Alloy ⅜ in. gage diameter.
[c] Tension specimen for S.A.E. 4130 Steel 0.187 in. gage diameter
[d] Oil quenched at 1625 F., Rockwell C 26.
[e] Oil quenched at 1625 F., Rockwell C 34.
[f] Oil quenched at 1625 F., Rockwell C 44.

complete curves, but it is believed that there are sufficient data to indicate the trend. Attention is called to a comparison of the curves for the rectangular and cylindrical specimens (heat-treated to Rockwell C 26 curve 1, Fig. 5). The curve for the rectangular specimens, if extended to the higher stresses of the round specimens, indicates a greater life for the former. Data, however, at such higher stresses for the rectangular specimens would no doubt somewhat flatten the sloping portion. In general, the data for the steel indicate lower fatigue characteristics for rectangular specimens. This is especially marked for the highest strength material.

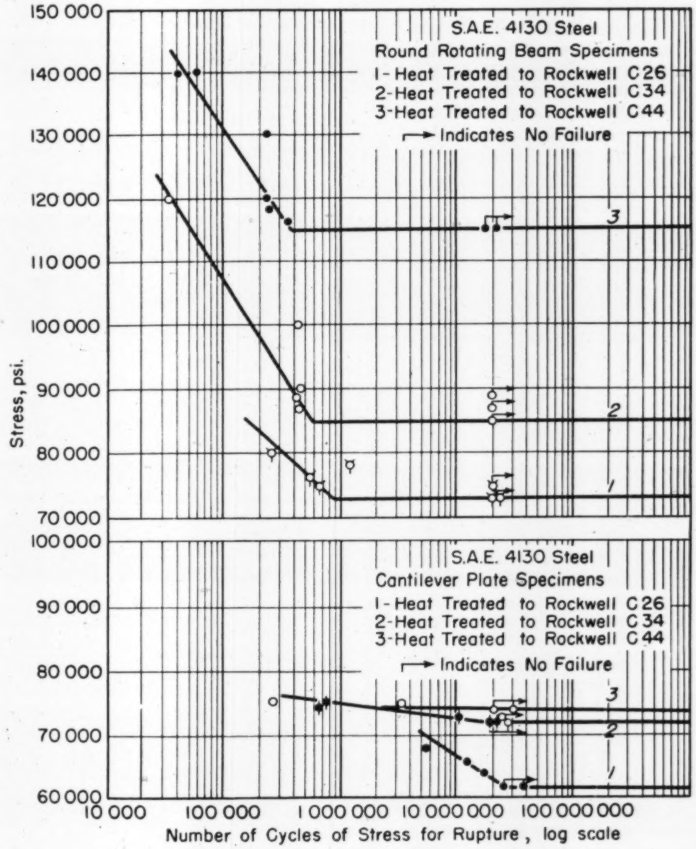


FIG. 5.—Fatigue Curves for S.A.E. 4130 Steel.

The data for the aluminum alloys, Figs. 6, 7, 8, and 9, would be expected to be subject to some scatter as the specimens were taken at different locations in cross-section and length of the extrusion. However, it is believed that the trend indicated by the average curves can be related to the type of test, irrespective of scatter. In Fig. 10 the data for an approximate minimum boundary curve faired through curves 1, 4, 3 for the round specimens would coincide with an approximate maximum curve faired through curves 1, 4, 2 for the rectangular specimens—thus indicating higher fatigue characteristics for the round specimens. In Fig. 6 the data for 75S-T show no appreciable difference except at the approximate endurance limit.

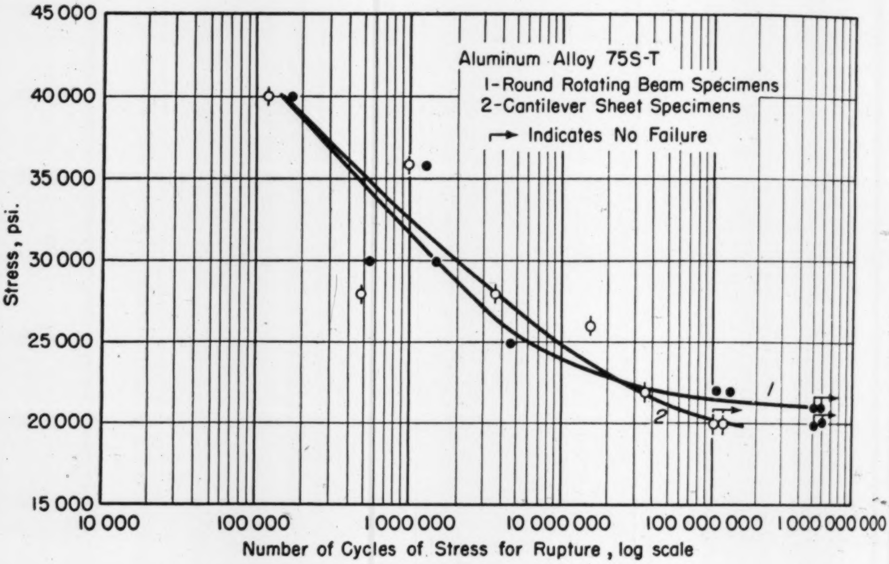


FIG. 6.—Fatigue Curves for Aluminum Alloy 75S-T.

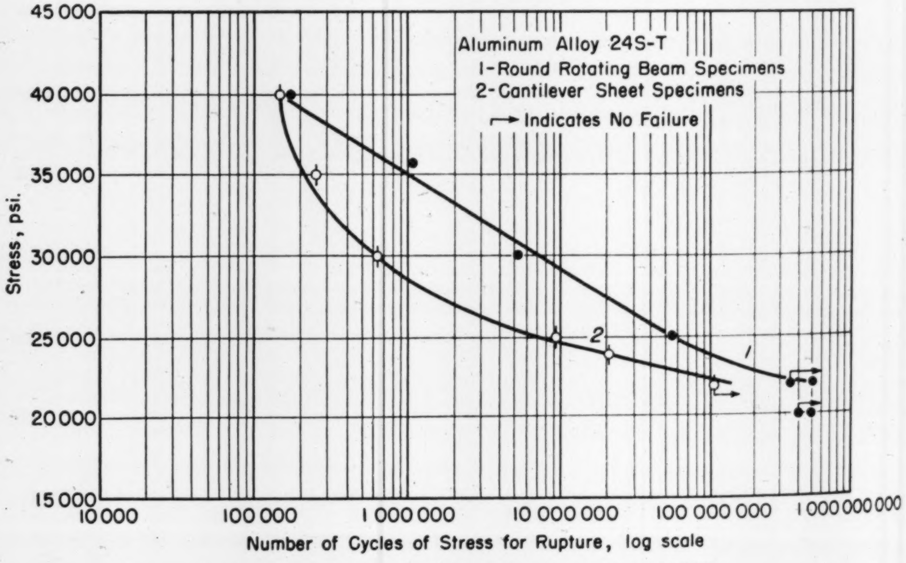


FIG. 7.—Fatigue Curves for Aluminum Alloy 24S-T.

CONCLUSIONS

It is believed that the findings indicate rather definite lower fatigue characteristics in reversed bending for a specific material tested in the Krouse plate machine in the form of rectangular specimens (nonrotating) compared with those of the cylindrical specimens (rotating) in the R. R. Moore machine.

obtained for rotating round specimens in the R. R. Moore machine (constant load) and both round and rectangular nonrotating specimens in the Krouse plate machine (fixed deflection).

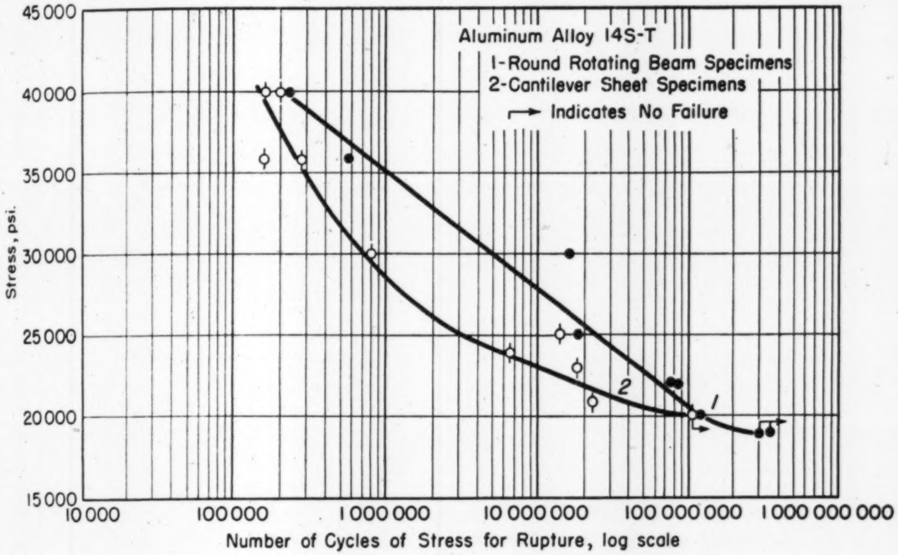


FIG. 8.—Fatigue Curves for Aluminum Alloy 14S-T.

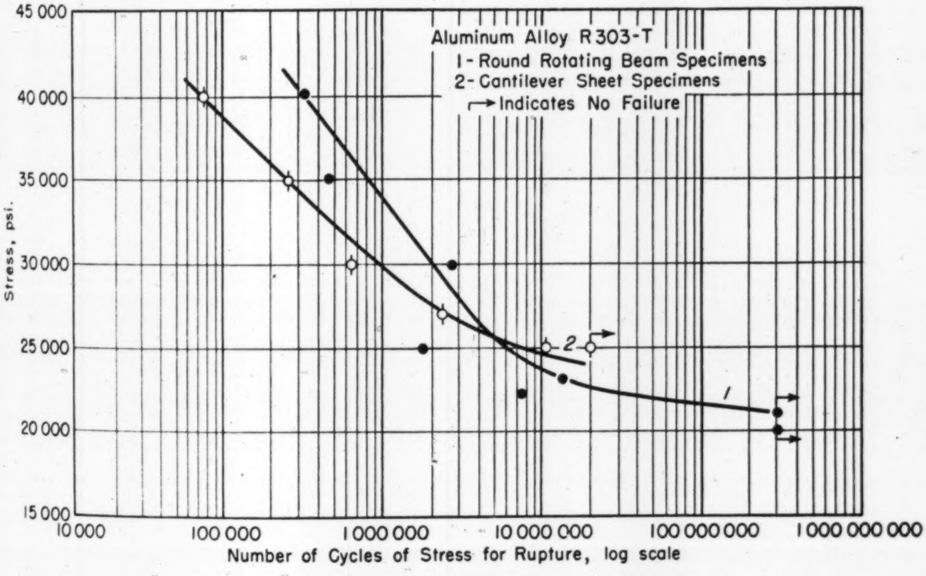


FIG. 9.—Fatigue Curves for Aluminum Alloy R303-T.

An investigation is now in progress in which data in reversed bending for material from a single plate are being

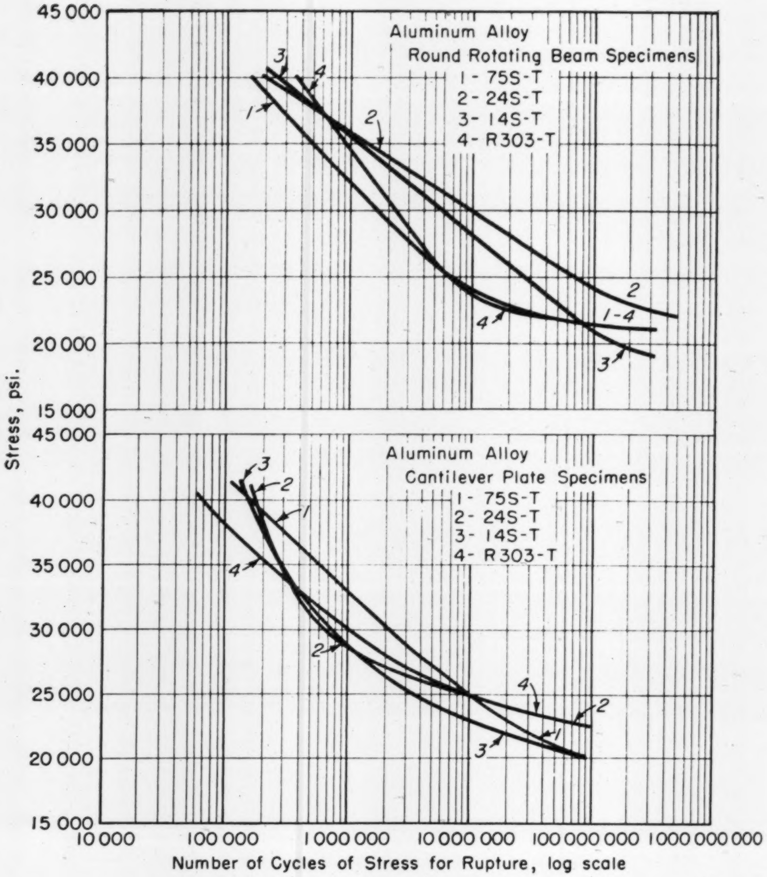


FIG. 10.—Fatigue Curves for Aluminum Alloys.

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(2) Thomas J. Dolan, "Certain Mechanical Strength Properties of Aluminum Alloys 25S-T and X76S-T," Nat. Advisory Comm. Aeronautics, Technical Note No. 914, October, 1943.

(3) H. F. Moore, "The Effect of Type of Testing Machine On Fatigue Test Results," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 133 (1941).

(4) L. R. Jackson, H. J. Grover, and R. C. McMaster, "Report on Fatigue Properties of Aircraft Materials and Structures," Battelle Memorial Inst. National Defense Research Committee of Office of Scientific Research and Development, 1 March, 1946.

DISCUSSION

MR. R. E. PETERSON[1] (*by letter*).—The data presented by Messrs. Fuller and Oberg are particularly valuable at this time in view of current discussions relative to standardizing of fatigue specimens. The data show that the results obtained are dependent on specimen design. In other laboratories similar results have been obtained, but these isolated cases have not been brought together in any systematic way. We should now give some thought to development of a theory consistent with the data, since if we are successful we will then be on safer ground in standardizing specimen dimensions.

As a beginning we might consider that failure is a matter of probability and that therefore the volume of highly stressed material is a significant parameter. For the purposes of comparison we could calculate the volume of material stressed to within an arbitrary percentage, η, of the peak stress. For making a calculation one could use, say, 5 per cent, but the actual value is not important as long as we are concerned with comparative values. Such calculations are not difficult. For the rotating beam, we have a band as shown in the accompanying Fig. 11. The inner surfaces can be approximated by conical surfaces and thus simplify calculation. Point B is determined sufficiently well by the section modulus relative to A. For the tapered flat specimens the corresponding regions will be essentially flat layers on the surfaces.

Without making calculations, it is apparent that the "95 to 100 per cent volume" is much larger in the flat steel specimen than in the rotating-beam steel specimen. The fatigue strength is considerably lower for the flat specimen.

On the other hand, it can be seen from Fig. 2 of the author's paper that the "95 to 100 per cent volume" is not so widely different for the aluminum alloy specimens. The difference in fatigue strengths is also not so great as for the steel specimens.

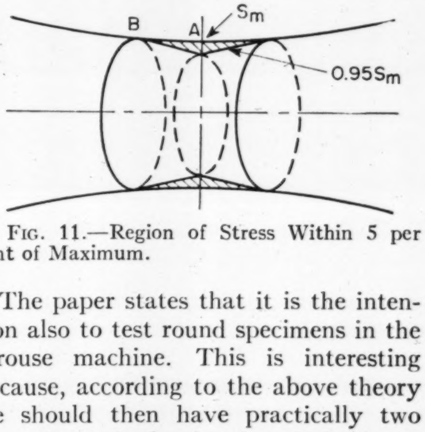

FIG. 11.—Region of Stress Within 5 per cent of Maximum.

The paper states that it is the intention also to test round specimens in the Krouse machine. This is interesting because, according to the above theory we should then have practically two spots instead of a band and the lower volume should result in a still higher fatigue strength.

Messrs. Fuller and Oberg have made available carefully obtained data and it is hoped that they will continue with the program and publish the results.

MR. THOMAS J. DOLAN[2] (*by letter*).—The data presented in this paper are of

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[1] Manager, Mechanics Div., Westinghouse Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

[2] Research Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

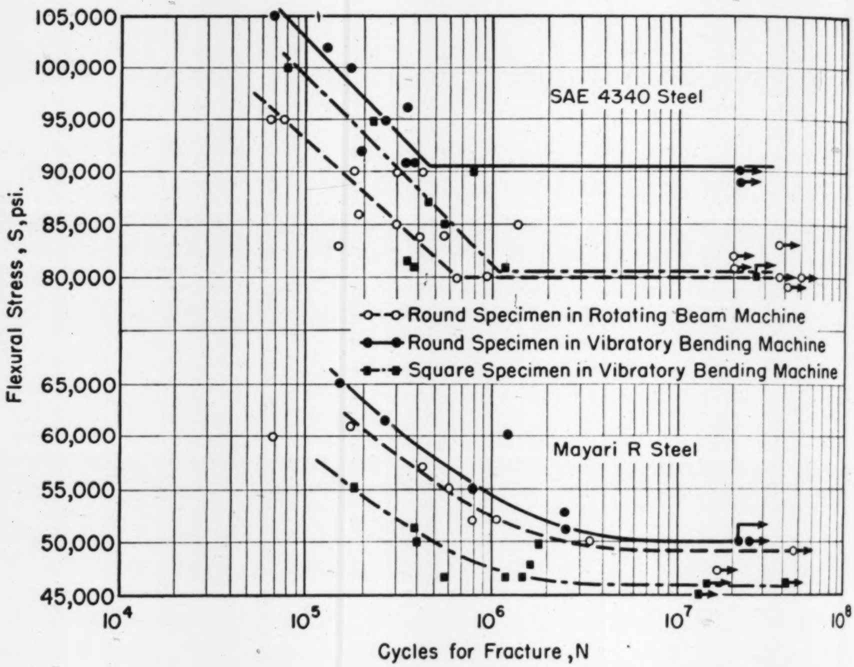

FIG. 12.—Influence of Shape of Cross-Section and of Type of Testing Machine on Flexural Fatigue Strength.

TABLE III.—MECHANICAL STRENGTH PROPERTIES OF SEVERAL METALS.

Material	Tensile Strength, psi.	Yield Strength in 0.2 per cent Offset, psi.	Elongation over 4 *D*, per cent	Depth of Specimen, in.	Fatigue Strength, psi., at 10 × 10^6 (cycles)	Fatigue Strength, psi., at 100 × 10^6 (cycles)	Fatigue Strength / Tensile Strength	Fatigue Strength / Fatigue Strength of Rotating Beam Specimens
ROUND SPECIMEN IN ROTATING BEAM MACHINE								
SAE 4340	150 400	142 200	18.2	0.30	80 000	...	0.531	...
Mayari R	75 200	52 600	33.2	0.30	49 000	...	0.652	...
25S-T Al. Alloy	55 000	36 600	24.7	0.26	24 000	19 000	0.346	...
X76S-T Al. Alloy	72 500	67 200	19.2	0.26	26 500	22 000	0.304	...
ROUND SPECIMENS IN VIBRATORY BENDING MACHINE								
SAE 4340	...	...	...	0.32	90 500	...	0.601	1.13
Mayari R	...	...	...	0.40	50 000	...	0.665	1.02
X76S-T Al. Alloy	...	...	...	0.26	27 500	24 000	0.331	1.09
RECTANGULAR SPECIMENS IN VIBRATORY BENDING MACHINE								
SAE 4340	...	...	...	0.32	80 500	...	0.535	1.006
Mayari R	...	...	...	0.40	46 000	...	0.612	0.940
25S-T Al. Alloy	...	...	...	0.25[a]	22 000	18 000	0.327	0.948
X76S-T Al. Alloy	...	...	...	0.24[a]	18 500	16 500	0.228	0.750
X76S-T Al. Alloy	...	...	...	0.25	20 000	18 500	0.255	0.840

[a] Specimens had a width three times the depth. All other "rectangular" specimens were square (width = depth).

especial interest to the writer since they show some of the same general trends as those observed in the previous work referred to by the authors.

The writer would like to supplement the paper with some additional test data that have recently been obtained to study the influence of the shape of cross-section on the endurance limits of two steels. The accompanying Fig. 12 shows flexural fatigue S-N curves for quenched and drawn S.A.E. 4340 steel and for normalized Mayari R steel (the Mayari R is a low alloy structural grade of steel). The static mechanical properties and fatigue strengths of these two steels are listed in the accompanying Table III together with some of the results from the previous tests (see references 1 and 2 of the paper) on aluminum alloys. These values of fatigue strength and static tensile strength have been plotted in block form in the accompanying Fig. 13 to facilitate comparison of the several results for each metal.

For tests in the vibratory bending type of fatigue machine only, these data indicate a fairly definite increase of fatigue strength for round specimens as compared with the values obtained from square (or rectangular) shapes. When one compares the fatigue strengths for *round* specimens only, the rotating-beam machine developed a somewhat lower fatigue strength than did the vibratory bending machine. However, the fatigue strengths of *rectangular* specimens in vibratory bending were found to be lower than those obtained from round rotating-beam specimens, except for the S.A.E. 4340 steel. It is not believed that any appreciable "size effect" existed in the tests of any one metal though the maximum depths of specimen varied slightly as shown in the accompanying Table III.

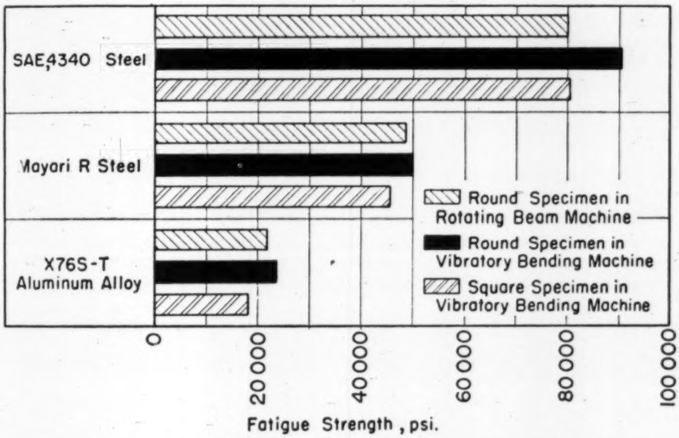

FIG. 13.—Effect of Testing Conditions on Fatigue Strength.

It would be of great interest to know what is the major cause of the differences observed in fatigue strength of the same metal under slightly different test conditions. When considering rotating-beam tests *versus* vibratory bending tests, the operating characteristics of the testing machines themselves may be factors affecting the results. There is always a tendency in the vibratory bending type of test for an unknown dynamic overthrow to occur (which may in some cases be appreciable) resulting in a somewhat smaller fatigue strength being computed. However, when both the round and square specimens were tested in the same vibratory bending type machine, these steels developed greater endurance limits in the *round* specimens. Similarly, the results of the previous tests on aluminum alloys indicated the same trend.

This difference in fatigue strength caused by a "shape" effect, does not seem logical and yet several possible explanations seem to be plausible. Reasoning from a statistical basis, there is less volume of metal in the region of the peak stress in the round specimens than in the square specimens, and hence a greater number of chances occur for a fiber of weaker strength to be in the extreme highly stressed condition that results in a fatigue failure. However, other recent unpublished tests still in progress on other shapes of cross-section do not seem to indicate this to be the complete explanation for a shape effect.

Consideration should be given to the probability that the residual stresses and localized strain hardening produced by machining operations are of importance and are likely to be altered by the differences in machining processes used to develop each shape of cross-section. That is, the square specimens are usually produced by milling with longitudinal tool cuts, whereas the round specimens are ordinarily produced by turning, using circumferential cuts with a single point tool in a lathe. The ordinary finishing and polishing methods customarily used on fatigue specimens of this type generally do not remove all of the cold-worked layers. Thus strain hardening and residual stresses of different magnitudes probably occur in the two shapes of specimens and influence the fatigue strengths of the final specimens to some (unknown) extent.

It is fairly well known that the extent to which plastic deformation progresses into a beam under static loads above that required to cause initial yielding is a function of the shape of the beam. It is possible that the minutely distributed localized plastic actions (that often accompany the development of progressive fractures) may also be influenced to some extent by the shape of cross-section of the beam (or the distribution of metal in the most highly stressed regions). A further study of some of these factors is now being made in connection with the tests of steels described above. It would be interesting to know if the authors have given this matter further study and have a plausible explanation for the differences in fatigue strength noted in the various types of specimens they tested.

MESSRS. F. B. FULLER AND T. T. OBERG (*authors' closure by letter*) The interest and discussions by Messrs. Peterson and Dolan are very much appreciated. They serve to emphasize the factors in the field of fatigue testing as related to types of specimen and testing machine. The authors feel that they are not in position to add any further comments at present. Data from tests in progress and the data and study being conducted by Mr. Dolan may aid in "shedding some light" on the problem of determining the fatigue properties of a material.

THE HIGH-TEMPERATURE FATIGUE STRENGTH OF SEVERAL GAS TURBINE ALLOYS[*]

BY P. R. TOOLIN[1] AND N. L. MOCHEL[2]

SYNOPSIS

One of the important properties of an alloy intended for use in the high-temperature components of a gas turbine is the fatigue strength at the service temperature. This paper gives some of the results of a program to determine the fatigue characteristics of many proposed gas turbine alloys at temperatures of 1200 F. and above. Results are given on alloys whose composition ranges from the older 18 per cent chromium, 8 per cent nickel type to the newer more highly alloyed materials. Both wrought and precision-cast (lost-wax process) materials were tested.

A brief description of the high-temperature fatigue machine used in this study is included.

As some of the high-temperature components of gas turbines are sometimes subjected to repeated stresses, the fatigue strengths of the alloys used in these components are a matter of importance. For this reason the high-temperature fatigue machines at the Westinghouse Research Laboratories have been devoted, for the past several years, almost exclusively to the testing of high-temperature alloys at temperatures of 1200 F. and above. Due to the shortage of equipment suitable for making these high-temperature fatigue tests, most of the tests described herein have been made as part of a program in cooperation with the War Metallurgy Committee. While many interested parties received periodic reports on this program, no compilation of the results has been available. This paper has been written to satisfy the requests for such a compilation and to make the information generally available.

The fatigue literature contains relatively little information on fatigue at temperatures of 1000 F. and higher, and the amount of this which deals with alloys of the types now being considered for application at temperatures of 1200 F. and over is very small. However, articles concerned with the fatigue of metals at temperatures of 1000 F. and higher have been listed in the references at the end of this paper.

METHOD OF TEST

Fatigue Machine:

Figure 1 shows the principal mechanical parts of the Westinghouse 120-cycle electromagnetic bending fatigue machine (**2**).[3] All fatigue tests reported herein were made in machines of this kind. The specimen is a fixed, non-rotating, cantilever which is vibrated in bending at a frequency of 120 cycles per sec. or roughly 10 million cycles in 24 hr. The

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Research Engineer, Mechanics Department, Westinghouse Research Laboratories, East Pittsburgh, Pa.

[2] Manager, Metallurgical Department, Westinghouse Electric Corp., Lester, Philadelphia, Pa.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 691.

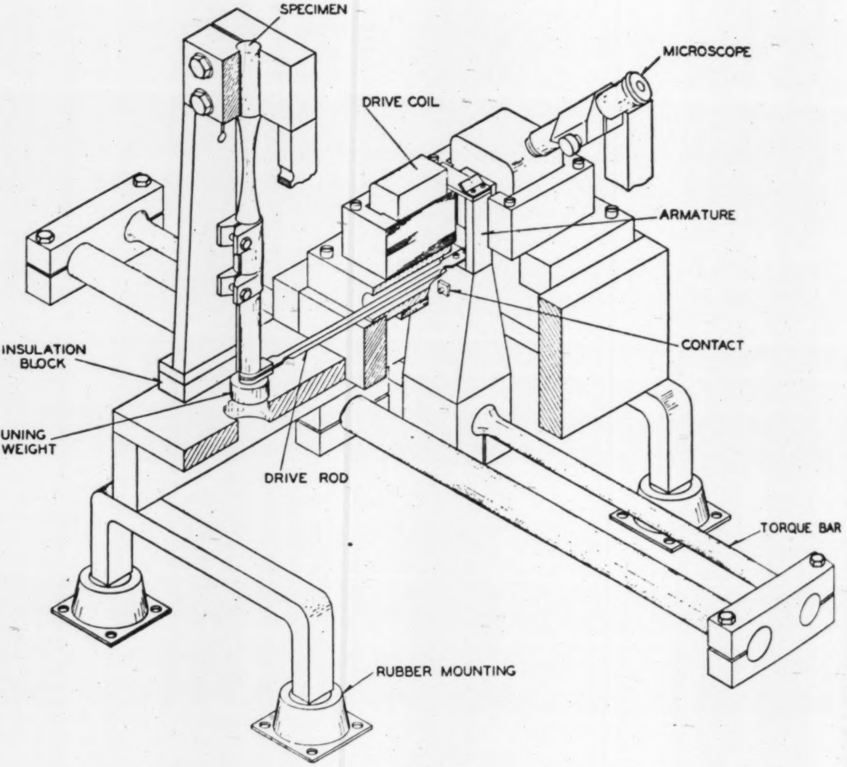
SPECIMEN
MICROSCOPE
DRIVE COIL
ARMATURE
CONTACT
INSULATION BLOCK
TUNING WEIGHT
DRIVE ROD
TORQUE BAR
RUBBER MOUNTING

FIG 1—Westinghouse 120-Cycle Fatigue Testing Machine.

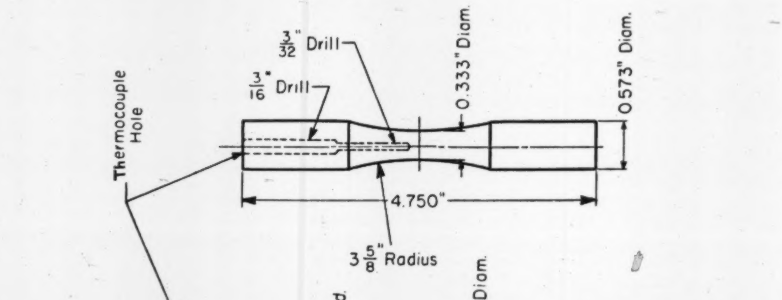
Thermocouple Hole
$\frac{3}{32}$" Drill
$\frac{3}{16}$" Drill
0.333" Diam.
0.573" Diam.
4.750"
$3\frac{5}{8}$" Radius
$\frac{1}{16}$" Rad.
$9\frac{1}{4}$" Rad.
0.550" Diam.
0.934" Diam.
0.934" Diam.
8.750"
$\frac{1}{4}$" Drill
$\frac{1}{8}$" Drill
14-D-7517

FIG. 2—Fatigue Specimens.

TABLE I.—ANALYSES.

Alloy Designation	Material	Chemical Composition, per cent															
		C	Mn	P	S	Si	Cr	Ni	Mo	Co	W	Cu	Cb	Ti	Al	Fe	N
GROUP 1—CHROMIUM-NICKEL-COBALT ALLOYS																	
101	Midvale GTA	0.19	0.65			0.64	19.91	32.10		30.05	5.41		4.00			bal.	(0.1)
102	S-816	0.36	0.72			0.19	18.40	20.23	3.72	45.63	4.23		3.04			bal.	
103	S-816	0.38	0.82			0.25	18.87	19.70	4.04	45.64	4.71		3.43			bal.	
104	Rolled Vitallium	0.22	0.65			0.53	27.42	2.28	5.53	62.20						0.70	
105	N-155, Low Carbon	0.15	1.46			0.42	20.84	20.58	3.18	20.48	2.26		1.06			bal.	0.14
106	S-590	0.47	1.35	0.013	0.013	0.82	19.40	19.07	4.03	19.26	4.00		3.98			bal.	
107	S-497	0.42	0.47	0.016	0.018	0.61	13.68	19.50	3.84	19.00	4.28		4.41			bal.	
108	Timken No. 2	0.14	1.56			0.92	17.64	27.65	9.52	35.60						bal.	
109	N-155, Low Carbon	0.06^a					20^a	20^a	3^a	20^a	2^a		1^a			bal.	0.15^a
110	N-155, High Carbon	0.36^a					20^a	20^a	3^a	20^a	2^a		1^a			bal.	0.15^a
111	N-155, Low Carbon	0.14	1.48			0.52	21.30	20.00	3.06	20.00	2.20		1.10			bal.	0.14
112	N-153	0.38	1.78			0.52	16.20	14.98	3.01	12.82	2.17		1.06			bal.	0.03
GROUP 2—NICKEL-MOLYBDENUM ALLOYS																	
201	Hastelloy-B	0.12	1.20			0.52	0.27	60.83	27.10							9.96	
202	Hastelloy-B	0.04	0.44			0.26		64.42	29.37							4.20	
GROUP 3—NICKEL-CHROMIUM AND NICKEL-CHROMIUM-COBALT ALLOYS TITANIUM AND ALUMINUM HARDENED																	
301	A.H. Inconel (X)	0.04	0.50		0.007	0.27	14.52	73.31				0.02	1.17	2.36	0.67	7.06	
302	A.H. Inconel	0.04	0.50		0.007	0.27	14.52	73.31				0.02	1.17	2.36	0.67	7.06	
303	80-20 A.H. Alloy	0.21	0.80	0.009	0.008	1.08	20.25	72.95		0.75		0.028		1.77	1.42	0.57	
304	K-42-B	0.05^a	0.7^a			0.59	18^a	42^a		22^a				2.17	0.48	bal	
305	A.H. Inconel (W)	0.04	0.73		0.010	0.52	15.02	73.98				0.07		2.42	0.49	6.70	
306	Nimonic-80	0.03	0.73			0.61	20.35	74.87						2.32	0.55	0.45	
307	Nimonic-80	0.38	0.40			0.54	22.38	72.56				0.13		2.19	1.24	0.28	
308	Refractaloy-26	0.05	0.65			0.72	17.9	37^a	2.80	20^a				2.60	0.30	bal.	
309	K-42-B	0.05				0.60	18^a	42^a		22^a				2.45	0.37	bal.	
GROUP 4—NICKEL-CHROMIUM-IRON ALLOYS																	
401	16-25-6	0.08	1.34	0.016	0.020	0.31	17.95	23.15	6.79							bal.	
402	S-495	0.49	0.36	0.012	0.014	0.81	13.75	19.03	4.43		2.83		4.15			bal.	
403	17, Low Carbon, Disk	0.10^a					13^a	20^a	0.60^a		2.25^a					bal.	
404	Hoskins 502	0.15	0.61	0.013	0.014	1.53	18.35	34.42								bal.	
405	GT-45	0.076	1.45	0.026	0.009	0.49	17.25	14.00	2.87			3.14	0.36	0.22		bal.	
406	17-W, High Carbon	0.46	0.58	0.016	0.013	0.43	13.19	19.16	0.52		2.48					bal.	
407	17-W, High Carbon	0.48	0.64	0.016	0.011	0.54	13.30	19.84	0.65		2.36					bal.	
408	Gamma Columbium	0.34	0.94	0.012	0.014	0.43	16.23	24.56	4.32				1.84			bal.	
409	25 Cr-20 Ni	0.23	1.03	0.011	0.012	1.80	24.66	19.88								bal.	
410	25 Cr-12 Ni	0.15	1.26			0.90	24.45	12.84	0.10							60.30	
GROUP 5—CHROMIUM-NICKEL-IRON ALLOYS																	
501	19-9-DL	0.27	0.72			0.60	18.50	9.20	1.32		1.41		0.49	0.18		bal.	
502	234A5	0.23	3.89		0.010	0.24	17.87	5.93	1.50		2.60		nil			bal.	
503	19-9-DL	0.33	1.14	0.016	0.015	0.65	19.10	9.05	1.35		1.14		0.35	0.16		bal.	
504	18-8-W-Mo	0.11	0.94	0.018	0.012	0.79	18.94	8.53	0.24		1.78					bal.	
505	19-9-W-Mo	0.26	0.48	0.016	0.010	0.51	19.26	8.66	1.40		1.20		0.37	0.27		bal.	
506	19-9-DL	0.29	1.41	0.022	0.008	0.50	18.59	9.04	1.19		1.40		0.45	0.29		bal.	
GROUP 6—PRECISION-CAST SPECIMENS																	
601	6059, Stoody	0.37				0.52	25.8	34.8	4.72	33.5						0.65	
602	X-40	0.45^a					28^a	11^a		53^a	7.5^a						
603	6059, Austenal	0.38				0.52	24.7	33.2	4.62	32.2						4.68	
604	Vitallium	0.25^a					28^a	2^a	6^a	64^a							
605	Vitallium	0.25^a					28^a	2^a	6^a	64^a							
606	61, Stellite 23	0.45^a					28^a	2^a		64^a	6^a						
607	6059	0.45^a					28^a	35^a	6^a	30^a							
608	Vitallium	0.15	0.28			0.46	27.07	3.08	5.27	bal.						1.20	
609	19-9-W-Mo	0.10^a					19^a	9^a	0.40^a		1.25^a		0.40^a			bal.	
610	S-495	0.45^a	1.00^a			0.50^a	14^a	20^a	4^a		4^a		4^a			bal.	
611	25-20	0.08	0.82	0.013	0.023	0.82	24.32	17.78	0.29	nil						bal.	
612	S-816	0.38	0.82			0.25	18.87	19.70	4.04	45.64	4.71		3.43			bal.	

[a] Type analysis

specimen is maintained at the desired testing temperature by means of a bell-type, resistance furnace which is placed over the specimen and its clamping fixtures. The driving mechanism consists of a cantilever beam, mounted on a torque bar, which is caused to vibrate by the a-c. electro-magnets denoted as drive coils in Fig. 1. As the entire vibrating system operates very close to resonance, the power input is relatively small.

Normally, the specimen is stressed in completely reversed bending, the mean stress equal to zero, and all tests reported herein were so made. However, by loading the specimen against the motor assembly, "range of stress" tests can be made.

Specimen:

Figure 2 shows the two sizes of specimens used. In Table III the specimen used for testing each alloy is given. Where possible, the specimens were provided with the thermocouple holes shown. However, in some cases the nature of the material made the drilling of these holes impractical. In these cases a thermocouple placed at the fixed end of the specimen was calibrated against the temperature of the critical section.

The geometry of the specimen and the nature of the loading place the critical section, point of maximum stress, slightly above the minimum diameter. All specimens, except the precision-cast ones with the sand-blasted as-cast surfaces, were polished in the region including the critical section. The final polishing was done in a longitudinal direction.

Due to the limited capacity of the precision-casting furnaces at the time of the start of this high-temperature program, all precision-cast specimens have been of the smaller size.

Materials:

As the field of high-temperature alloys is still in its infancy, the composition of the materials tested covers a considerable range. In the hope of achieving better order, the alloys have been divided—in some cases quite arbitrarily—into six groups. These alloy groups have been designated as follows: group 1, chromium-nickel-cobalt; group 2, nickel-molybdenum; group 3, nickel-chromium and nickel-chromium-cobalt, hardened by titanium and aluminum; group 4, nickel-chromium-iron; group 5, chromium-nickel-iron; and group 6, alloys that have been precision cast (lost-wax process). Group 6 differs from the other groups in that it was assembled on a processing procedure basis rather than on a composition basis. Consequently, it contains alloys of rather dissimilar chemical analyses, some of which appear in their wrought form in the other groupings.

The materials are listed in their respective groups in Table I, which gives the chemical composition of the alloys. For cross reference between the various tables and curves the alloys have been given a numerical designation—those in group 1 receiving the numbers 101, 102, etc., those in group 2 the numbers 201, 202, etc., and so on. In general, the alloys in any one group have been numbered according to their 1200 F. fatigue strength at 100 million cycles, the best alloy receiving the lowest number. In Table III, the Westinghouse test designation number has also been given so that those who have received previous Westinghouse high-temperature fatigue reports may easily identify them.

Group 1, Chromium-Nickel-Cobalt Alloys, is composed of alloys having approximately 20 per cent chromium; S-495 with 14 per cent and rolled Vitallium with 27 per cent are the extreme variations. With the exception of the 2 per cent nickel content of rolled Vitallium, the nickel content is within the range from 15 to 32 per cent with the majority having very close to

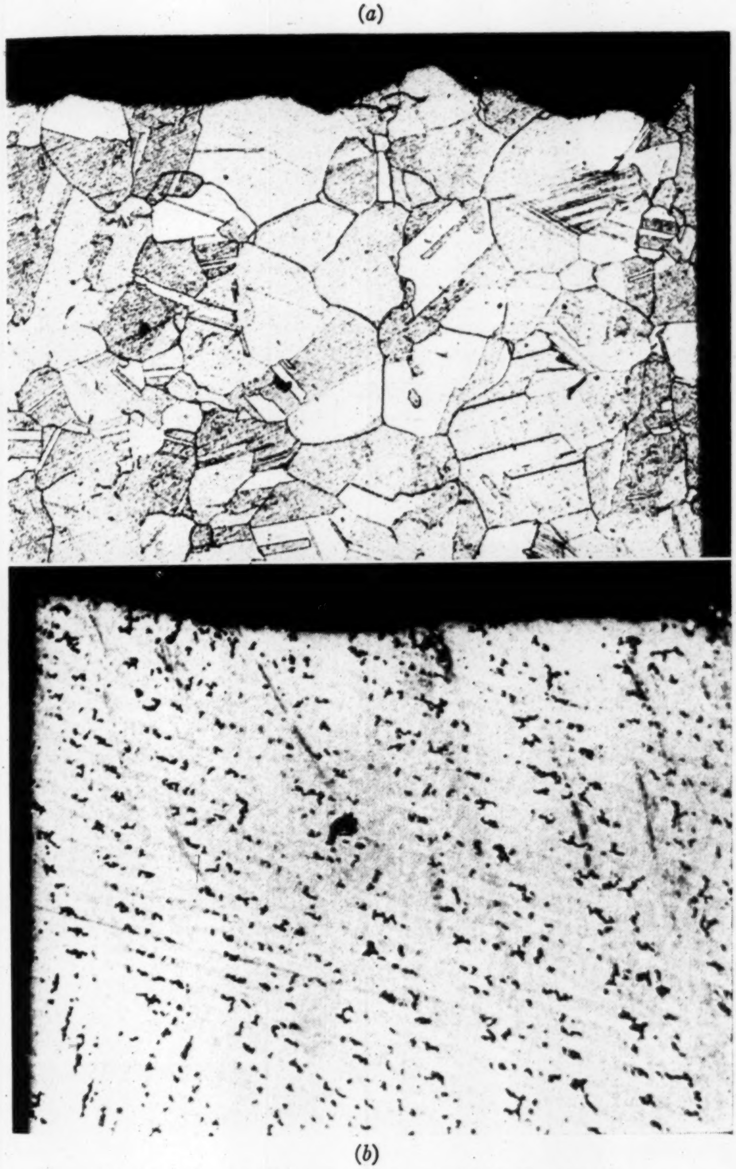

FIG. 3—Fatigue Failures of Wrought K-42-B and Cast Vitallium from Specimens Tested at 1200 F. (x 100).

(*a*) K-42-B (304). 8.9 Million Cycles at 66,000 psi.
(*b*) Cast Vitallium (605). 3.5 Million Cycles at 54,000 psi.

20 per cent. The percentage of cobalt varies from 13 per cent for N-153 to 62 per cent for rolled Vitallium. In addition, these alloys contain from 1 to 5 per cent of one or all of the following: molybdenum, tungsten, and columbium. Timken No. 2 is an exception with 9 per cent tungsten only.

Group 2, Nickel-Molybdenum Alloys, consists of only one alloy, Hastelloy-B. This alloy is singular because of its high nickel and molybdenum content and its complete lack of chromium.

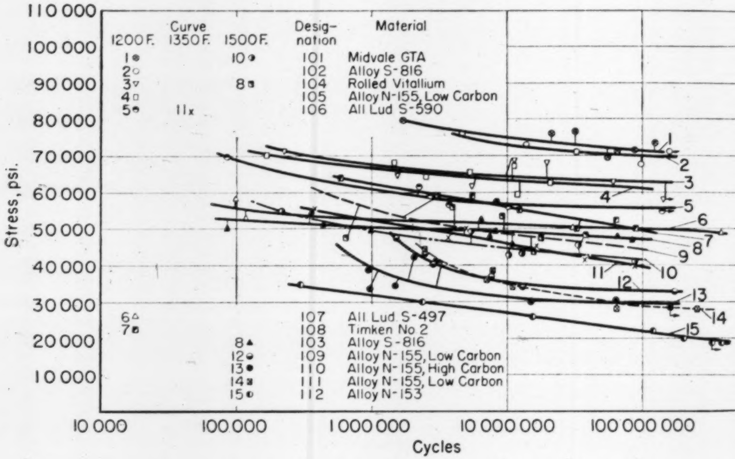

FIG. 4—*S-N* Diagrams, Chromium-Nickel-Cobalt Alloys at 1200 F. and 1500 F.

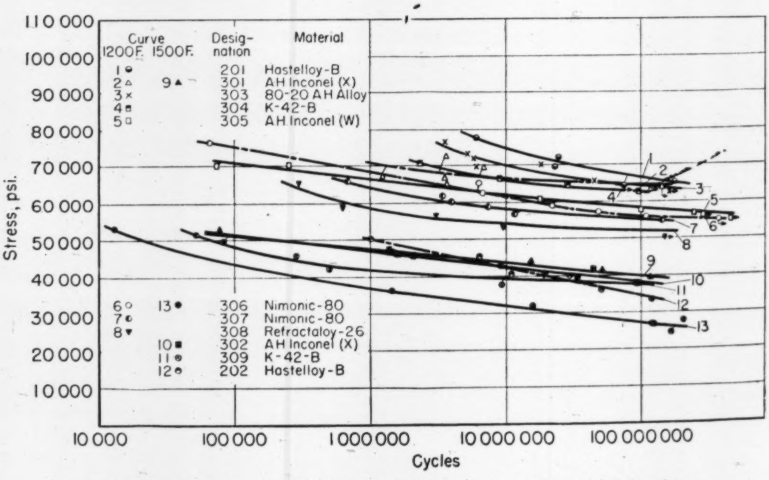

FIG. 5—*S-N* Diagrams, Nickel-Molybdenum and Nickel-Chromium and Nickel-Chromium-Cobalt Alloys (Titanium and Aluminum Hardened) at 1200 F. and 1500 F.

Group 3, Nickel-Chromium and Nickel-Chromium-Cobalt Alloys, is made up of alloys containing up to 2.6 per cent titanium and 1.4 per cent aluminum. Refractaloy-26 and K-42-B both have approximately 18 per cent chromium, 40 per cent nickel, and 20 per cent cobalt with the former also having about 3 per cent molybdenum. The other alloys are very similar to each other, all having from 15 to 20 per cent chromium and 70 to 75 per cent nickel. The presence or absence of an appreciable percentage of iron, the fact that the Inconel X contains columbium, and a fair variation in carbon are the only appreciable differences. All, with the exception of the 80-20 A.H. alloy and one of the Nimonic-80's, are low in carbon content.

Group 4, Nickel-Chromium-Iron Alloys, is a "catch-all" for the various iron alloys containing up to 25 per cent each of chromium and nickel, but not falling into the 18-8-W-Mo class. Hoskins-502 with 34 per cent nickel is the only exception to the above.

Group 5, Chromium-Nickel-Iron Alloys,

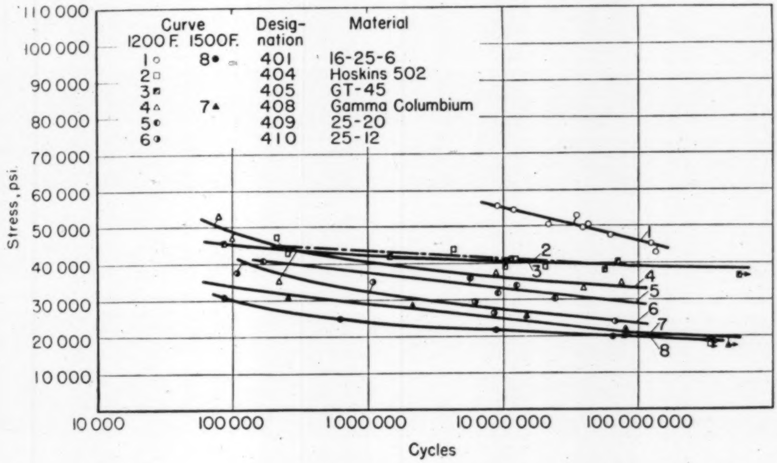

Fig. 6—*S-N* Diagrams, Nickel-Chromium-Iron Alloys at 1200 F. and 1500 F.

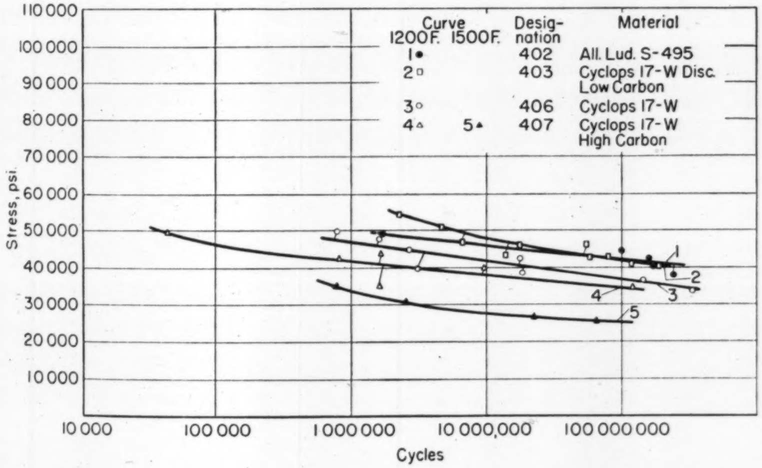

Fig. 7—*S-N* Diagrams, Nickel-Chromium-Iron Alloys at 1200 F. and 1500 F.

consists of the 18-8-W-Mo type alloys, some of which have columbium and titanium additions.

Group 6, the Precision-Cast Specimens, contains all alloys which were tested in the precision-cast form. This has made for a rather wide variation in analyses. The absence of titanium hardened alloys is due to the nature of the process and the affinity of this element for oxygen.

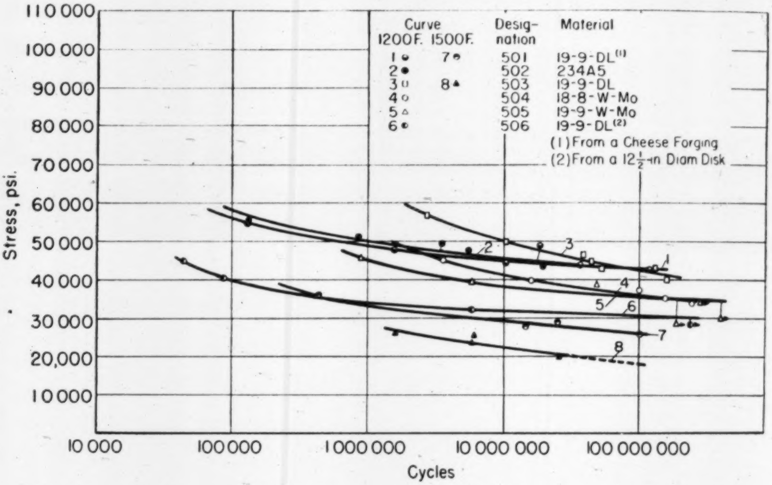

FIG. 8—*S-N* Diagrams, Chromium-Nickel-Iron Alloys at 1200 F. and 1500 F.

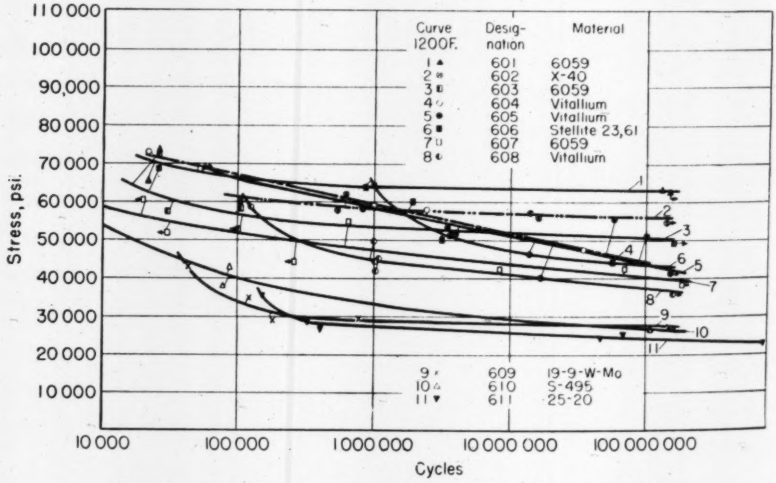

FIG. 9—*S-N* Diagrams, Precision Cast Specimens at 1200 F.

Heat Treatment:

The heat treatment, bar size of the stock from which the endurance specimens were made, and the source of the material are given in Table II. It seems appropriate at this point to emphasize: (1) that the alloys were tested with these specific heat treatments, and (2) that in many cases the applied treatments may not be those giving maximum fatigue properties.

All precision-cast specimens were tested in the as-cast condition. However, as the specimens came from several different suppliers, each having his own casting procedure, there is a chance for considerable structural variation. This comes principally from variations in the mold temperatures and in the pouring temperatures.

Fatigue Test Results:

The fatigue test data and resulting *S-N* diagrams are shown in Figs. 4 to 10, inclusive. With the exception of group 2, the curves are all by group. The curves for Hastelloy-B, group 2, are shown in Fig. 5 with the nickel-chromium and nickel-chromium-cobalt alloys.

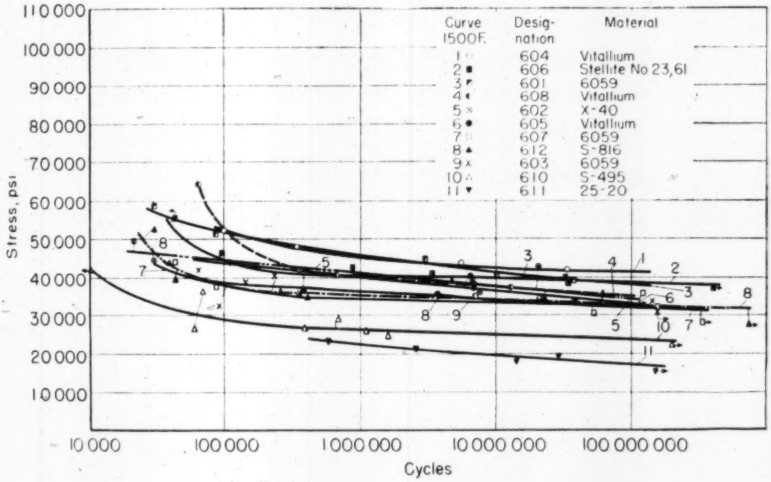

FIG. 10—*S-N* Diagrams, Precision Cast Specimens at 1500 F.

In general, an attempt has been made in this program to secure an endurance curve which is well fixed at 100 million cycles. This number of cycles was chosen because, in most cases, at this point the high-temperature endurance curve, determined under completely reversed stress, approaches a straight line on the plot of stress *versus* the logarithm of the number of cycles to failure. Many of the curves still have appreciable slope at more than 100 million cycles, for which reason it is important to state the number of cycles at which a high-temperature fatigue strength is given.

A word of caution must be given in regard to the fatigue curves for the precision-cast alloys. As these specimens were tested in the as-cast condition, some of the materials "aged" during the test. In these cases, this resulted in increasingly strong materials as the tests progressed, which in turn undoubtedly resulted in some of the endurance curves being much flatter than would be the case had the materials been more stable at the test temperatures. The 1200 F. curve of 6059 alloy, designation 601, is an example of this. This alloy also provides an indication of the part casting procedure can play in determining fatigue strength. The materials designated by numbers 601 and 603 are practically identical in analysis. The 601 material was cast in a relatively cold mold compared to that used for the 603 material, which procedure resulted in the 601 specimens having a much finer structure than the 603 specimens. At 1200 F. the fine-structured material has a fatigue strength

TABLE II.—HEAT TREATMENT AND SOURCE OF ALLOYS.

Alloy Designation	Material	Heat Treatment		Bar Size	Supplier
		Solution Treatment	Aging or Stress Relief Treatment		
		GROUP 1—CHROMIUM-NICKEL-COBALT ALLOYS			
101......	Midvale GTA	For 1200 F. test: As rolled For 1500 F. test: 1500 F.—4 hr., A.C.[a]		1 in. diam.	The Midvale Co.
102......	S-816	2300 F.—1 hr.—W.Q.[a]	1400 F.—16 hr.—A.C.	1 in. diam.	Allegheny-Ludlum Steel Corp. (Submitted by the General Electric Co.)
103......	S-816	2350 F.—1 hr.—W.Q.	1500 F.—16 hr.—A.C.	1 in. sq.	Allegheny-Ludlum Steel Corp.
104......	Rolled Vitallium	2250 F.—1 hr.—A.C.	1400 F.—6 hr.—A.C.	1⅛ in. diam.	Crucible Steel Co.
105......	N-155, Low carbon	None	1200 F.—50 hr.—A.C.	1 in. sq.	Union Carbide and Carbon Co.
106......	S-590	2275 F.—40 min. W.Q.	1400 F.—16 hr.—A.C.	1$\frac{1}{16}$ in. diam.	Allegheny-Ludlum Steel Corp.
107......	S-497	For 1200 F. test: 2000 F.—1 hr.—W.Q. For 1500 F. test: 2250 F.—1 hr.—W.Q.	1300 F.—16 hr.—A.C. 1500 F.—16 hr.—A.C.		
108......	Timken No. 2	Hot rolled	1600 F.—2 hr.—A.C.	1⅛ in. diam.	Timken Roller Bearing Co.
109......	N-155, Low carbon	None	1500 F.—50 hr.—A.C.	1 in. sq.	Submitted by the War Metallurgy Committee
110......	N-155, High carbon	2200 F.—1 hr.—W.Q.	1500 F.—50 hr.—A.C.	1 in. sq.	
111......	N-155, Low carbon	None	1500 F.—50 hr.—A.C.	1 in. sq.	
112......	N-153	Preheated 1550 F.—30 min. 2200 F.—30 min.—O.Q.[a]	1500 F.—50 hr.—A.C.	1¼ in. sq.	
		GROUP 2—NICKEL-MOLYBDENUM ALLOYS			
201......	Hastelloy-B	W.Q. from 2000 F.	1200 F.—4 hr.—F.C.[a]	1 in. diam.	Haynes Stellite Co.
202......	Hastelloy-B	2050 F.—20 min.—A.C.	1900 F.—24 hr.—A.C.		Haynes Stellite Co. (Submitted by the General Electric Co.)
		GROUP 3.—NICKEL-CHROMIUM AND NICKEL-CHROMIUM-COBALT ALLOYS (TITANIUM AND ALUMINUM HARDENED)			
301......	A.H. Inconel (X)	2100 F.—24 hr.—W.Q.	1300 F.—16 hr.—A.C.		International Nickel Co.
302......	A.H. Inconel (X)	2100 F.—1 hr.—W.Q.	1300 F.—16 hr.—A.C.		
303......	80-20 A.H. Alloy	2000 F.—1 hr.—W.Q.	1350 F.—20 hr.—F.C.	1⅛ in. sq.	Carpenter Steel Co.
304......	K-42-B	1950 F.—1 hr.—W.Q.	1350 F.—20 hr.—A.C.	1⅛ in. diam.	Westinghouse Elec. Corp.
305......	A.H. Inconel (W)	1975 F.—6 hr.—W.Q.	1300 F.—16 hr.—A.C.	1 in. diam.	International Nickel Co.
306......	Nimonic-80	For 1200 F. test: 1950 F—4 hr.—W.Q. For 1500 F. test: 1950 F.—4 hr.—W.Q.	1300 F.—20 hr.—A.C. 1500 F.—50 hr.—A.C.	1 in. diam.	
307......	Nimonic-80	1950 F.—?—W.Q.	1292 F.—16 hr.—A.C.	1¼ in. diam.	British Air Ministry
308......	Refractaloy-26	2100 F.—1 hr.—O.Q.	1350 F.—20 hr.—A.C.	$\frac{13}{32}$ in. diam.	Westinghouse Elec. Corp.
309......	K-42-B	2100 F.—1 hr.—O.Q.	1500 F.—20 hr.—A.C.	1 in. diam.	
		GROUP 4—NICKEL-CHROMIUM-IRON ALLOYS			
401......	16-25-6	For 1200 F. test: W.Q. from 2000 F. For 1500 F. test: 2150 F.—45 min.—W.Q.	1200 F.—4 hr.—F.C. 1500 F.—50 hr.—A.C	1 in. diam.	Timken Roller Bearing. Co.
402......	S-495	For 1200 F. test: 2000 F.—1 hr.—W.Q.	1300 F.—16 hr.—A.C.	1 in. diam.	Allegheny-Ludlum Steel Co.
403......	17, Low carbon Disk			Disk	Submitted by the Army Air Corps.
404......	502	W.Q. from 2000 F.	1200 F.—4 hr.—F.C.	1 in. diam.	Hoskins Mfg. Co.
405......	G.T.-45	2250 F.—30 min.—W.Q.	1200 F.—5 hr.—A.C.	1$\frac{3}{32}$ in. diam.	Rustless Iron & Steel Co.
406......	17-W, High carbon	W.Q. from 2000 F.	1200 F.—4 hr.—F.C.	1⅛ in. sq.	Universal Cyclops Steel Co.

[a] A.C. = air cooled.
W.Q. = water quenched.
F.C. = furnace cooled.
O.Q. = oil quenched.

TABLE II—*Continued.*

Alloy Designation	Material	Heat Treatment[a]		Bar Size	Supplier
		Solution Treatment	Aging or Stress Relief Treatment		
		GROUP 4—NICKEL-CHROMIUM-IRON ALLOYS—*Continued*			
407......	17-W, High carbon	Preheat at 1500 F. Hot Work at 2100 F. Solution Treatment: 2100 F.—1 hr.—A.C. Equalized at 1200 F. Cold work: 12½% at 1200 F. Stress Relief: 1200 F.—4 hr.—A.C.			Crucible Steel Co. (Submitted by the General Elec. Co.)
408......	Gamma columbium	For 1200 F. test: 1950 F.—4 hr.—W.Q.	1500 F.—50 hr.—A.C.		The Midvale Co.
		For 1500 F. test: 2250 F.—45 min.—O.Q.	1500 F.—50 hr.—A.C.	1⅛ in. diam.	
409......	25 Cr-20 Ni	W.Q. from 2000 F.	1200 F.—4 hr.—F.C.	1¾ in. diam.	Edgecomb Steel Co.
410......	25 Cr-12 Ni	W.Q. from 2000 F.	1200 F.—4 hr.—F.C.	1⅛ in. diam.	Rustless Iron & Steel Co.
		GROUP 5—CHROMIUM-NICKEL-IRON ALLOYS			
501......	19-9-DL cheese forging	Preheat at 1500 F.; hot work at 2100 F. Sol. Treat. 2100 F.—1 hr.—A.C. Equalized at 1200 F.; cold work at 1200 F.—12½%; strain relief—anneal—1200 F.—4 hr.—A.C.			Crucible Steel Co. (Submitted by the General Elec. Co.)
502......	234A5	Stress relief: 1200 F.—4 hr.—furnace cooled forged—2100 F.		1¼ in. diam.	Crucible Steel Co.
503......	19-9-DL	Solution treated—2100 F.—1 hr.—A.C. Cold work—15% at 1200 F. Anneal—1200 F.—4 hr.—A.C.			Universal Cyclops Steel Co. (Submitted by the General Elec. Co.)
504......	18-8-W-Mo	Stress relieved: 1200 F.—4 hr.—furnace cooled		1¾ in. diam.	Universal Cyclops Steel Co.
505......	19-9-W-Mo	Stress relieved: 1200 F.—50 hr.—A.C.		1 in. diam.	
506......	19-9-DL 12½″ diam. disk, 3¼″ thick	Stress relieved: 1200 F.—8 hr.—A.C.		Disk	
		GROUP 6—PRECISION-CAST SPECIMENS			
601......	6059	As cast by the Stoody process			General Electric Co.
602......	X-40, Stellite No. 31	As cast			Haynes Stellite Co.
603......	6059	As cast by the Austenal process			General Electric Co.
604......	Vitallium	As cast			B. F. Hirsch Inc.
605......	Vitallium	As cast			Austenal Laboratories
606......	61, Stellite No. 23	As cast			Haynes Stellite Co.
607......	6059	As cast			Haynes Stellite Co.
608......	Vitallium	As cast by the Austenal process			General Electric Co.
609......	19-9-W-Mo	As cast			Haynes Stellite Co.
610......	S-495	As cast			Austenal Laboratories
611......	25-20	As cast			Austenal Laboratories
612......	S-816	As cast			B. F. Hirsch, Inc.

of 63,000 psi. as compared to 51,000 psi. for the coarser-structured specimens.

The fatigue failures of the wrought materials proceeded, in general, in a transcrystalline manner. Photomicrographs of sections through the fractures of a wrought K-42-B specimen and of a precision-cast Vitallium specimen are shown in Fig. 3.

The 100 million cycle fatigue strengths at 1200 F. and 1500 F., the room temperature hardness before test, and the size of the specimen used are given in Table III. This table shows that there is considerable variation in endurance strengths in each group and, of course, large variation among the groups. In general, in any specific group, the alloys having the higher before-test hardness show superior endurance properties. Due to the instability of some of the as-cast precision-cast specimens at the test

TABLE III.—ENDURANCE STRENGTHS, HARDNESS AND SPECIMEN SIZE.

Alloy Designation	Westinghouse Test Designation	Material	Fatigue Strength at 100 Million Cycles, psi.		Room Temperature Hardness Before Testing[a]	Specimen
			1200 F.	1500 F.		
		GROUP 1—CHROMIUM-NICKEL-COBALT ALLOYS				
101.....	X-720	Midvale GTA	71 000	42 000	372 DPH[b]	L[e]
102.....	S-816	S-816	70 000	47 000	315 DPH	L
103.....	X-662	S-816		47 000	328 DPH[c]	L
612.....	X-678	Cast S-816		32 000	274 DPH	S
104.....	X-721	Rolled Vitallium	63 000	44 000	362 DPH	S
604.....	X-637	Cast Vitallium	44 000	41 000	357 DPH	S
105.....	J-518	N-155, Low Carbon	61 000		27 R_c	L
106.....	X-757	S-590	55 000	40 000[d]	303 DPH	L
107.....	X-621	S-497	50 000		218 DPH[c]	L
108.....	X-756	Timken No. 2	50 000		279 DPH	L
109.....	NR-66	N-155, Low Carbon		33 000	280 DPH	S
110.....	NR-21B	N-155, High Carbon		30 000	294 DPH	S
111.....	X-645	N-155, Low Carbon		29 000	280 DPH	L
112.....	NR-19	N-153		22 000	236 DPH	L
		GROUP 2—NICKEL-MOLYBDENUM ALLOYS				
201.....	X-603	Hastelloy-B	66 000		232 BHN	L
202.....	Q-34	Hastelloy-B		35 000	308 DPH	L
		GROUP 3—NICNEL-CHROMIUM AND NICKEL-CHROMIUM-COBALT ALLOYS (TITANIUM AND ALUMINUM HARDENED)				
301.....	X-715	A.H. Inconel (X)	65 000	40 000	340 DPH	L
302.....	X-716	A.H. Inconel (X)		38 000	315 DPH	L
303.....	X-753	80-20 A.H. Alloy	64 000		377 DPH	L
304.....	X-657	K-42-B	63 000		330 DPH	L
305.....	X-632	A.H. Inconel (W)	58 000		338 DPH	L
306.....	X-628	Nimonic-80	56 000	27 000	331 DPH[f] 252 DPH[g]	L
307.....	X-672	Nimonic-80	55 000		325 DPH	L
308.....	KB-1024	Refractaloy-26	52 000		329 DPH	S
309.....	K-55	K-42-B	52 000	38 000	285 DPH	L
		GROUP 4—NICKEL-CHROMIUM-IRON ALLOYS				
401.....	X-620	16-25-6	46 000	20 000	250 DPH[f] 384 DPH[g]	L
402.....	X-618	S-495	42 000		223 DPH	L
610.....	X-643	S-495, Cast	27 000	23 000	281 DPH	S
403.....	X-649	17, Low Carbon, Disk	42 000		302 DPH	S
404.....	X-605	Hoskins 502	39 000		192 DPH	L
405.....	X-780	GT-45	39 000		254 DPH	L
406.....	X-612	17-W, High Carbon	37 000		287 DPH	L
407.....	A-763	17-W, High Carbon	34 000	25 000	320 DPH	L
408.....	X-630	Gamma Columbium	33 000	21 000	225 DPH	L
409.....	X-617	25 Cr-20 Ni	29 000		206 DPH	L
611.....	X-761	25 Cr-20 Ni, Cast	24 000	17 000	166 DPH	S
410.....	X-602	25 Cr-12 Ni	23 000		215 DPH	L
		GROUP 5—CHROMIUM-NICKEL-IRON ALLOYS				
501.....	A-762	19-9-DL	43 000	26 000	314 DPH	L
502.....	X-659	234A5	43 000		280 DPH	L
503.....	X-656	19-9-DL	42 000	18 000	360 DPH	L
504.....	X-614	18-8-W-Mo	36 000		228 DPH	L
505.....	X-635	19-9-W-Mo	36 000		251 DPH	L
609.....	X-639	19-9-W-Mo, Cast	27 000		23 R_D	S
506.....	T-40812-4	19-9-DL	30 000		26 R_D	S
		GROUP 6—PRECISION-CAST SPECIMENS				
601.....	6059	6059 (Stoody Process)	63 000	38 000	24 R_c	C[h]
602.....	X-726	X-40, Stellite 31	56 000	34 000	293 DPH	C
603.....	WLA-121	6059 (Austenal Process)	51 000	32 000	34 R_D	C
604.....	X-637	Vitallium (Hirsch Process)	44 000	41 000	357 DPH	G
605.....	X-642	Vitallium	44 000	33 000	345 DPH	G
606.....	X-638	61 (Stellite 23)	44 000	38 000	338 DPH	G
607.....	X-644	6059 (Stellite 27)	41 000	32 000	250 DPH	G
608.....	WLA-339	Vitallium	38 000	35 000	41 R_D	C
609.....	X-639	19-9-W-Mo	27 000		23 R_D	G
610.....	X-643	S-495	27 000	23 000	281 DPH	G
611.....	X-761	25-20	24 000	17 000	166 DPH	C
612.....	X-678	S-816		32 000	38 R_D	C

[a] DPH = Diamond pyramid hardness.
BHN = Brinell hardness number.
R_c = Rockwell hardness "C" scale.

[b] 50-kg. Vickers.

[c] 30-kg. Vickers.

[d] At 1350 F.

[e] L and S indicate respectively the 0.550 in. and 0.333 in. minimum diameter specimens.

For 1200 F. test specimens.

[g] For 1500 F. test specimens.

[h] All precision cast specimens were the small size. C denotes an as cast surface with a sand blasting. G denotes ground surface.

temperatures, the before-test hardness of these alloys has less significance.

The twelve alloys showing the best 1200 F. and 1500 F. fatigue strengths at 100 million cycles are listed in Tables IV and V, respectively. In compiling these tables, alloys which are very similar, such as the A.H. Inconels, 80-20 A.H. alloy, and Nimonic-80 have been considered as one alloy. At 1200 F. the endurance strengths of these twelve alloys vary from 71,000 psi. for Midvale GTA alloy to 50,000 psi. for Timken No. 2 alloy. The improvement in properties afforded by these alloys becomes apparent if the above values are compared with the fatigue strength of 8000 psi. at 1200 F. and 10 million cycles (tests made at 2500 stress cycles per min.) reported by Moore and Alleman (9) for a 0.17 per cent carbon steel.

TABLE IV.—COMPOSITION AND FATIGUE STRENGTHS OF ALLOYS TESTED AT 1200 F. (TWELVE HIGHEST).

Alloy Designation	Material	Chemical Composition, per cent														Fatigue Strength at 100 Million Cycles, psi.	
		C	Mn	Si	Cr	Ni	Mo	Co	W	Cu	Cb	Ti	Al	Fe	N	1200 F.	1500 F.
101	Midvale GTA	0.19	0.65	0.64	19.91	32.10	....	30.05	5.41	....	4.00	....	....	bal.	(0.15)	71 000	42 000
102	S-816	0.36	0.72	0.19	18.40	20.23	3.72	45.63	4.23	....	3.04	....	....	bal.	....	70 000	47 000
201	Hastelloy-B	0.12	1.20	0.52	0.27	60.83	27.10	....	....	....	....	....	....	9.96	....	66 000	35 000^b
301	A.H. Inconel (X)	0.04	0.50	0.27	14.52	73.31	....	....	....	0.02	1.17	2.36	0.67	7.06	....	65 000	40 000
304	K-42-B	0.05^a	0.7	0.59	18^a	42^a	....	22^a	....	....	....	2.17	0.48	....	....	63 000	....
104	Rolled Vitallium	0.22	0.65	0.53	27.42	2.28	5.53	62.20	....	....	....	....	....	0.70	....	63 000	44 000
601	6059, Cast	0.37	....	0.52	25.8	34.8	4.72	33.5	....	....	....	....	....	0.65	....	63 000	38 000
105	N-155, Low-Carbon	0.15	1.46	0.42	20.84	20.58	3.18	20.48	2.26	....	1.06	....	....	bal.	0.14	61 000	33 000^c
602	X-40, Cast	0.54	1.02	0.61	25.46	10.27	0.21	50.28	6.74	....	....	....	....	....	....	56 000	34 000
106	S-590	0.47	1.35	0.82	19.40	19.07	4.03	19.26	4.00	....	3.98	....	....	bal.	....	55 000	....
308	Refractaloy-26	0.05	0.65	0.72	17.9	37^a	2.80	20^a	....	....	....	2.60	0.30	bal.	....	52 000	....
108	Timken No. 2	0.14	1.56	0.92	17.64	27.65	9.52	35.60	....	....	....	....	....	bal.	....	50 000	....

^{a} Type analysis.
^{b} For designation No. 202.
^{c} For designation No. 109.

TABLE V.—COMPOSITION AND FATIGUE STRENGTHS OF ALLOYS TESTED AT 1500 F. (TWELVE HIGHEST).

Alloy Designation	Material	Chemical Composition, per cent														Fatigue Strength at 100 Million Cycles, psi.	
		C	Mn	Si	Cr	Ni	Mo	Co	W	Cu	Cb	Ti	Al	Fe	N	1500 F.	1200 F.
102	S-816	0.36	0.72	0.19	18.40	20.23	3.72	45.63	4.23	....	3.04	....	....	bal.	....	47 000	70 000
104	Rolled Vitallium	0.22	0.65	0.53	27.42	2.28	5.53	62.20	....	....	....	....	....	0.70	....	44 000	63 000
101	Midvale GTA	0.19	0.65	0.64	19.91	32.10	....	30.05	5.41	....	4.00	....	....	bal.	....	42 000	71 000
604	Vitallium, Cast	0.25^a	....	....	28^a	2^a	6^a	64^a	....	....	....	....	....	....	....	41 000	44 000
301	A.H. Inconel (X)	0.04	0.50	0.27	14.52	73.31	....	....	....	0.02	1.17	2.36	0.67	7.06	....	40 000	65 000
606	61, Stellite 23, Cast	0.45^a	....	....	28^a	2^a	....	64^a	6^a	....	....	....	....	....	....	38 000	44 000
309	K-42-B	0.05	....	0.60	18^a	42^a	....	22^a	....	....	....	2.45	0.37	bal.	....	38 000	52 000
601	6059, Cast	0.37	....	0.52	25.8	34.8	4.72	33.5	....	....	....	....	....	0.65	....	38 000	63 000
202	Hastelloy-B	0.04	0.44	0.26	....	64.42	29.37	....	....	....	....	....	....	4.20	....	35 000	66 000^b
602	X-40, Cast	0.54	1.02	0.61	25.46	10.27	0.21	50.28	6.74	....	....	....	....	....	....	34 000	56 000
109	N-155, Low Carbon	0.06^a	....	....	20^a	20^a	3^a	20^a	2^a	....	1^a	....	....	bal.	0.15	33 000	61 000^c
612	S-816, Cast	0.38	0.82	0.25	18.87	19.70	4.04	45.64	4.71	....	3.43	....	....	bal.	....	32 000	

^{a} Type analysis.
^{b} For designation No. 201.
^{c} For designation No. 105.

Table IV shows that the compositions of these twelve alloys vary over an extensive range. This indicates that other factors besides composition may have considerable effect. Evidence of this is given by the better than 80,000 psi., 100 million cycle, 1200 F. fatigue strength obtained on Refractaloy-26 in a program now in progress to study the major metallurgical factors affecting high-temperature strength. The 52,000 psi. fatigue strength listed for Refractaloy-26 was obtained on material which had received the optimum treatment for high creep strength at 1500 F. It thus appears that maximum fatigue and creep properties do not necessarily go hand in hand.

At 1500 F., the order is somewhat different than at 1200 F. but the large variation in the compositions, Table V, of the better alloys still exists. The highest fatigue strength reported is 47,000 psi. for S-816 and the twelfth alloy from this is precision cast S-816 with a fatigue strength of 32,000 psi. While there are only two precision-cast materials in the first twelve at 1200 F., at 1500 F. there are five. A comparison of the rolled and precision-cast Vitallium is of interest. At 1200 F. the rolled material has a fatigue strength of 63,000 psi. compared to 44,000 psi. for the cast, while at 1500 F. the values are 44,000 psi. and 41,000 psi., respectively. Similarly, in the cases of the other alloys—S-816, S-495, 19-9-W-Mo and 25-20—which have been tested in both the wrought and cast form, the wrought form gives the higher fatigue strengths. Cross (8) obtained similar results on wrought and sand cast 18 per cent chromium, 8 per cent nickel alloy tested at room temperature and at 1200 F.

SUMMARY AND CONCLUSIONS

This paper has presented information obtained from more than seventy fatigue curves at 1200 and 1500 F. on various high-temperature alloys tested in bending under completely reversed stress.

While the 71,000 psi. fatigue strength at 1200 F. and 100 million cycles of Midvale GTA is the highest fully reported herein, mention was made of a better than 80,000 psi. value obtained for Refractaloy-26 in a program now in progress. The 47,000 psi. fatigue strength for S-816 is the highest value reported at 1500 F.

At 100 million cycles the fatigue curves of most of the alloys tested approach a straight line on the standard semilogarithmic *S-N* diagram. However, the slope of this straight part of the diagram varies considerably from alloy to alloy.

The wrought form of a given alloy appears to give higher fatigue strength at 1200 to 1500 F. than does the "as precision-cast" form.

Observation of the analyses of the better alloys indicates that relatively high fatigue strength can be obtained with alloys of rather wide composition. It is pointed out that other factors besides composition exercise a considerable effect.

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(15) H. F. Moore and T. M. Jasper, "An Investigation of the Fatigue of Metals," University of Illinois, Engr. Expr. Station, *Bulletin No. 152* (1925).

(16) F. C. Lea, "The Effect of Low and High Temperatures on Materials," *Proceedings*, Inst. Mechanical Engrs., p. 1051 (1924).

(17) F. C. Lea and H. P. Budgen, "The Effect of High Temperature on the Range of Repetition Stresses on Steel," *Engineering*, Vol. 10, Oct. 3, 1924, p. 500; Oct. 10, 1924, p. 532.

(18) "Engineering Properties of Inconel," *Bulletin T-7*. The International Nickel Co. Inc., New York, N. Y.

DISCUSSION

Mr. H. F. Moore.[1]—The authors have presented an excellent fatigue study for their material for turbine service—service in which the frequency of application of cycles of stress is high. As has been pointed out, in the range of the temperatures which are being considered both creep and fatigue are acting. The action of fatigue seems to be, in general, a function of number of cycles of stress; the action of creep is a function of time; so that it would be quite possible that the rate of application of load might be a quite vital factor. Over in the soft metals, such as lead, it is extremely important, since results can be changed three or four times by the changes of speed of testing of cycles.

I wanted to mention this, not as a criticism of the paper which seems to me to be an excellent one for the purpose, but simply because when we get into these high temperatures we must remember that both creep and fatigue are acting, but they do not act the same, that is, depending on the same variable.

Mr. H. H. Bleakney.[2]—The question of fatigue at high temperatures is one which is of great concern to designers of gas turbines today.

The temperatures at which gas turbine blades are currently operating are in the creep range of the materials employed. The available evidence indicates that, at these temperatures, the allowable creep stresses, and even the stresses for rupture in 1000 hr. are lower than the endurance stresses for the materials employed. The question then presents itself: Is the fatigue value really significant or is not the stress to rupture, being lower, the controlling factor? An expression of the authors' opinion on this point would be welcome.

The speed of the fatigue testing machine may also be significant. Mr. Moore has shown that for lead the fatigue values obtained at high testing speeds are higher than those obtained at lower speed. Since the gas turbine blade alloys tested display plasticity at the testing temperatures, it is not unlikely that the same speed effect might be a factor. There is some evidence that it is.

Mr. H. J. Grover.[3]—I should like to ask the authors one question—whether the appearance of failure varies with the temperature in any of these extensive tests that they have reported?

Mr. P. R. Toolin (*author's closure*).—Messrs. Moore and Bleakney have both raised the very pertinent question of how important a factor the frequency of stressing is in endurance tests conducted at the temperatures reported. In the case of alloys with poor oxidation resistance, one would expect that, at least at the high stress end of the S-N diagram, tests made at high speed would give superior values to those made at low speeds. Moore and Alleman's tests (reference 9 of paper) on a 0.17 per cent carbon steel at 800 and 1200 F. give credence to this hypothesis. For the more highly alloyed materials, H. C. Cross has reported (reference 8 of paper) fatigue tests made on a 19 per cent chromium, 10 per cent nickel steel at 1200 F. where the values obtained at 200 rpm. were superior to those obtained at 2500 rpm. This is at variance with the generally accepted trend for room temperature fatigue. G. N. Krouse's work[4] indicates that while the speed effect at room temperature is relatively insignificant below 10,000 rpm., at higher speeds the endurance strength increases with increasing speed. Moore's work on lead,[5] as was mentioned in the discussion, indicates higher endurance strengths with higher testing speeds. This may be indicative of what can be expected of blading alloys at elevated temperatures, but in my opinion only a properly controlled investigation will satisfactorily answer this question. We plan to make such an investigation.

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[2] National Research Council of Canada, Ottawa, Ont., Canada.

[3] Assistant Supervisor, Battelle Memorial Inst., Columbus, Ohio.

In regard to Mr. Bleakney's question on the relative importance of fatigue strength and stress-rupture strength, it would seem that whether a turbine blade fails in fatigue or stress-rupture would be a function of both the properties of the blading material and of the design—that is, it is dependent upon the nature of the stress which the design imposes upon the blade. The stress in a turbine blade consists of both a steady and a variable component, and the ratio of the magnitudes of these two component stresses is a function of the service and the design. If the magnitude of the variable-stress component is large in respect to that of the steady component, and the frequency is high—which it usually is—the failure will probably be in fatigue. Similarly, if the steady component is much the larger the failure will be of the stress-rupture type. Of course, most failures undoubtedly should be attributed to range-of-stress loading, a field which is sorely in need of further investigation. The fact that the variable-stress component is somewhat more subject to control than is the steady component may, in the future, tend to place most of the failures at the stress-rupture end of the range-of-stress diagram.

In reply to Mr. Grover, there has been no definite change noted in the appearance of the fatigue fractures in the temperature range from 1200 to 1500 F.

I wish to thank the discussers for their remarks.

MR. N. L. MOCHEL (*author's closure by letter*).—Mr. Bleakney has raised the question as to relative importance of fatigue strength and stress-rupture properties. This same question was raised informally by several others, in connection with the paper. It is not surprising that this question should arise. In actual practice on jet engines, there have been blade failures of both types. Some have been pure creep-rupture failures, an accidental combination of too high temperature for the normal tension stresses involved, with the cross-section of the blade showing marked reduction of cross-sectional area. Other failures have been observed in which there was progressive fracture as distinct as any well-recognized fatigue failure. There have been some failures that one finds it difficult, from the fracture, to be positive as to whether it is of one type or another. They are probably of a combined nature.

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[4] G. N. Krouse, "A High Speed Fatigue Testing Machine and Some Tests of Speed Effect on Endurance Limit," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 156 (1934).

[5] H. F. Moore, B. B. Betty, and C. W. Dollins, "Creep and Fracture of Lead and Lead Alloys," University of Illinois, Engr. Expr. Station, *Bulletin No. 272* (1935).

There is a research project being organized at the present time to study the relation between fatigue and static strength of materials for gas turbines, and this work will improve our knowledge on the question above involved.

[See also discussion of Gohn and Ellis paper, p. 722]

FATIGUE CHARACTERISTICS OF SOME COPPER ALLOYS*

By H. L. Burghoff[1] and A. I. Blank[1]

Synopsis

This paper describes the fatigue characteristics of copper and several copper alloys, available commercially in the form of rod, as determined in rotating-beam fatigue tests. Fatigue properties are given for more than one temper in several instances. Tests were performed on two types of specimens, as polished and as notched.

The ratios of fatigue strength for polished specimens to tensile strength of the materials included in the investigation range from 0.26 to 0.52. The effects of notching on fatigue properties are influenced by notch radius and degree of working of the metal in the notching operation. Notch sensitivity increases with notch sharpness. Work-hardening of the metal adjacent to the notch may offset very considerably the loss of fatigue strength due to notching. The grain size and manner of occurrence of the alpha and beta phases in naval brass and manganese bronze influence the fatigue properties of these alloys; high fatigue strength is associated with fine grain structures, whereas considerably lower fatigue strength is associated with coarse, acicular structures. Fatigue strength of hard-drawn silicon bronze increases with the degree of final reduction.

An extensive program of rotating beam fatigue tests has been performed on a number of copper-base rod materials. Both polished and notched specimens were tested in order to determine the inherent fatigue properties of the test materials and also to provide some indication of the effect of stress concentration on those properties.

Materials

The materials, as listed in Table I, include two types of copper, cartridge brass, three leaded brasses, naval brass, manganese bronze, two silicon bronzes, an age-hardenable copper-nickel-phosphorus-tellurium alloy, and cupri-nickel, 30 per cent. Rotating-beam tests on polished specimens of copper-base materials have been reported by Anderson and Smith[2] and by Anderson, Swan, and Palmer.[3] Except for a few alloys of similar composition, the alloys and tempers and types of specimens included in the present work differ from those of the earlier investigations.[2,3]

In evaluating the fatigue strength of a material, proper recognition should be given to such factors as heat treatment, microstructure, extent of working and size, as well as chemical composition, since fatigue properties change in accordance with variations of all these factors. Each of the materials tested in this investigation has therefore been described as completely as possible in Table I in order to provide a sound basis for comparison of the relative merits of the various materials with regard to

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Research Metallurgist and Assistant Research Metallurgist, respectively, Chase Brass and Copper Co., Waterbury, Conn.

[2] A. R. Anderson and Cyril Stanley Smith, "Fatigue Tests on Some Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 849 (1941).

[3] A. R. Anderson, E. F. Swan, and E. W. Palmer, "Fatigue Tests on Some Additional Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 678 (1946).

TABLE I.—SUMMARY OF RESULTS OF TENSION AND FATIGUE TESTS.

Material and Composition	Diameter of Rod as Processed, in.	Temper	Tension Tests				Fatigue Tests						
			Stress at 0.2 per cent Offset, psi.	Stress at 0.5 per cent Elongation, under load, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Type of Fatigue Specimen	Notch Radius, in.	Apparent Work-Hardening Adjacent to Notch	Fatigue Strength at 1,000,000 Cycles, psi.	Fatigue Strength at 100,000,000 Cycles, psi.	Ratio, Fatigue Strength to Tensile Strength	Fatigue Strength Reduction Factor
Electrolytic copper Cu 99.93	0.625	Drawn 30 per cent (0.040-mm. grain size)	40 000	41 000	44 000	19.5	Polished	...	...	25 000	16 500	0.37	...
			...	...	...	...	Notched	0.0015	Considerable	18 000	12 000	...	1.4
Tellurium copper Cu 99.10 (by diff.), Te 0.52, P 0.02, Zn 0.36	0.625	Drawn 17 per cent (0.060-mm. grain size)	37 000	37 500	39 500	25.5	Polished	...	...	24 000	16 500	0.42	...
			...	...	...	...	Notched	0.002	None	17 000	12 500	...	1.3
Cu-Ni-P-Te alloy Cu 98.13, Ni 1.16, P 0.21, Te 0.50, Pb<0.004, Fe<0.01, Zn <0.02	0.530	Quenched, drawn 39 per cent and aged (0.065-mm. grain size)	72 500	71 700	81 800	14.0	Polished	...	...	44 000	31 500	0.39	...
			...	...	...	...	Notched	0.015	None	28 700	22 500	...	1.4
			...	...	...	...	Notched	0.0006	None	30 000	20 000	...	1.6
Cu-Ni-P-Te alloy Cu 98.05, Ni 1.15, P 0.22, Te 0.51, Pb 0.01, Fe 0.01, Zn 0.05	1.000	Quenched, drawn 29 per cent and aged (0.065-mm. grain size)	71 500	70 500	81 400	16.5	Polished	...	...	42 500	36 000	0.44	...
			...	...	...	...	Notched	0.0003	None	21 100	14 500	...	2.5
Cu-Ni-P-Te alloy Cu 98.29, Ni 0.99, P 0.19, Te 0.46, Pb<0.01, Fe 0.015, Zn 0.05	2.375	Quenched, drawn 18 per cent and aged (0.050 mm. grain size)	53 500	53 300	66 900	42.5	Polished	...	...	35 700	26 000	0.39	...
			...	...	...	...	Notched	0.0003	None	22 300	13 000	...	2.0
Cartridge brass Cu 69.37, Fe 0.015, Pb<0.01, Zn 30.61 (by diff.)	0.625	Drawn 21 per cent (0.120-mm. grain size)	45 500	45 800	57 000	42.0	Polished	...	...	31 300	15 000	0.26	...
			...	...	...	...	Notched	0.0025	Considerable	30 500	20 500	...	0.8
Cupro-nickel, 30 per cent Cu 68.04, Ni 30.67, Mn 0.53, Fe 0.49, Zn 0.17 Pb<0.05	0.560	Drawn 33 per cent (0.025-mm. grain size)	77 000	75 500	81 700	12.0	Polished	...	...	42 800	34 500	0.42	...
			...	...	...	...	Notched	0.015	None	24 300	16 000	...	2.2
			...	...	...	...	Notched	0.0006	Very Slight	23 500	15 000	...	2.3
Leaded commercial bronze Cu 88.77, Pb 2.00, Mn 0.03, Fe 0.01, Zn 9.20 (by diff.)	0.625	Drawn 17 per cent (0.040-mm. grain size)	39 500	40 500	45 000	27.5	Polished	...	...	26 000	20 000	0.44	...
			...	...	...	...	Notched	0.0015	None	19 000	17 000	...	1.2
Free-cutting brass Cu 61.78, Pb 3.31, Fe 0.10, Zn 34.81 (by diff.)	0.625	Drawn 20 per cent (0.040-mm. grain size)	42 500	41 000	55 000	28.0	Polished	...	...	30 500	20 000	0.36	...
			...	...	...	...	Notched	0.002	None	22 000	17 000	...	1.2
Forging brass Cu 61.34, Pb 1.87, Fe 0.04, Zn 36.75 (by diff.)	0.531	Annealed at 1200 F. (0.035-mm. grain size)	16 700	17 000	49 200	51.0	Polished	...	...	24 000	14 000	0.28	...
			...	...	...	...	Notched	0.005	Considerable	26 500	25 000	...	0.6
	0.531	Drawn 28 per cent (0.035-mm. grain size)	58 500	55 200	70 600	11.0	Polished	...	...	39 500	27 500	0.39	...
			...	...	...	...	Notched	0.004	Considerable	37 000	18 000	...	1.5

Naval brass Cu 59.88, Sn 0.68, Pb 0.09, Fe 0.02, Ni 0.02, Zn 39.31 (by diff.)	0.500	Drawn 24 per cent, fine grain	72 500	65 500	87 000	14.0	Polished	...	...	48 000	33 000	0.40	...
Cu 59.74, Sn 0.71, Pb 0.06, Fe 0.08, Ni 0.015, Zn 39.39 (by diff.)	0.500	Drawn 24 per cent, coarse grain	68 800	61 000	87 300	9.0	Polished	...	...	29 000	21 000	0.26	...
Cu 60.53, Sn 0.68, Pb 0.12, Fe 0.02, Ni 0.01, Zn 38.64 (by diff.)	0.530	Drawn 27 per cent, fine grain	76 500	68 000	87 200	12.5	Polished	...	...	48 000	31 500	0.36	...
			...	...	...	...	Notched	0.0006	None	22 800	15 000	...	2.1
Cu 60.37, Sn 0.75, Pb 0.08, Fe 0.02, Ni 0.01, Zn 38.77 (by diff.)	0.530	Drawn 27 per cent, coarse grain	79 000	69 500	91 400	11.5	Polished	...	...	41 800	26 500	0.29	...
			...	...	...	...	Notched	0.0006	None	27 400	15 000	...	1.8
Manganese bronze Cu 58.21, Fe 1.21, Sn 0.78, Mn 0.08, Pb 0.09, Zn 39.63 (by diff.)	0.525	Drawn 28 per cent, fine grain	72 000	58 000	89 600	12.0	Polished	...	...	42 500	28 000	0.31	...
			...	...	...	...	Notched	0.0035	Considerable	39 000	21 500	...	1.3
	0.525	Drawn 28 per cent, coarse grain	73 500	61 500	89 100	12.0	Polished	...	...	39 500	24 500	0.27	...
			...	...	...	...	Notched	0.0025	Considerable	40 000	19 000	...	1.3
1.5 per cent silicon bronze Cu 97.18, Si 1.43, Pb 0.025, Fe 0.01, Zn 1.35 (by diff.)	0.560	Annealed, 0.025-mm. grain size	10 800	11 400	40 600	57.0	Polished	...	...	20 400	15 000	0.37	...
			...	...	...	...	Notched	0.015	None	12 700	9 000	...	1.7
			...	...	...	...	Notched	0.001	None	11 000	7 000	...	2.1
Cu 97.12, Si 1.46, Pb<0.01, Fe 0.03, Zn 1.39 (by diff.)	0.625	Drawn 21 per cent (0.020-mm. grain size)	48 000	48 500	54 000	29.5	Polished	...	...	33 000	28 000	0.52	...
			...	...	...	...	Notched	0.0015	Considerable	28 000	24 000	...	1.2
Cu 97.18, Si 1.43, Pb 0.25, Fe 0.01, Zn 1.35 (by diff.)	0.560	Drawn 37 per cent (0.030-mm. grain size)	59 000	58 500	63 600	15.5	Polished	...	...	36 700	28 500	0.45	...
			...	...	...	...	Notched	0.015	None	21 800	15 000	...	1.9
			...	...	...	...	Notched	0.001	None	20 600	10 500	...	2.7
3 per cent silicon bronze Cu 95.88, Si 3.01, Fe 0.045, Pb 0.01, Zn 1.05 (by diff.)	0.563	Drawn 10 per cent (0.020-mm. grain size)	42 400	45 400	62 700	49.5	Polished	...	...	29 500	24 000	0.38	...
			...	...	...	...	Notched	0.015	None	20 900	14 000	...	1.7
			...	...	...	...	Notched	0.001	None	16 000	10 000	...	2.4
	0.563	Drawn 23 per cent (0.035-mm. grain size)	56 700	54 000	76 600	29.0	Polished	...	...	36 500	28 000	0.37	...
			...	...	...	...	Notched	0.015	None	20 900	14 000	...	2.0
			...	...	...	...	Notched	0.001	None	15 300	11 000	...	2.5
	0.563	Drawn 33 per cent (0.025-mm. grain size)	67 000	58 000	93 200	18.5	Polished	...	...	41 800	29 000	0.31	...
			...	...	...	...	Notched	0.015	None	23 700	18 000	...	1.6
			...	...	...	...	Notched	0.001	None	15 200	10 500	...	2.8
	0.563	Drawn 44 per cent (0.040-mm. grain size)	80 000	62 000	102 500	14.0	Polished	...	...	45 200	34 500	0.33	...
			...	...	...	...	Notched	0.015	None	23 000	17 500	...	1.9
			...	...	...	...	Notched	0.001	None	14 600	12 000	...	2.8
Aluminum bronze Cu 87.84, Al 9.65, Fe 1.95, Te 0.52, Si 0.03, Pb 0.005, Zn<0.02	0.565	Drawn 10 per cent	53 000	50 500	93 000	19.0	Polished	...	...	46 000	28 500	0.31	...
			...	...	...	...	Notched	0.0015	Slight	47 000	20 000	...	1.4

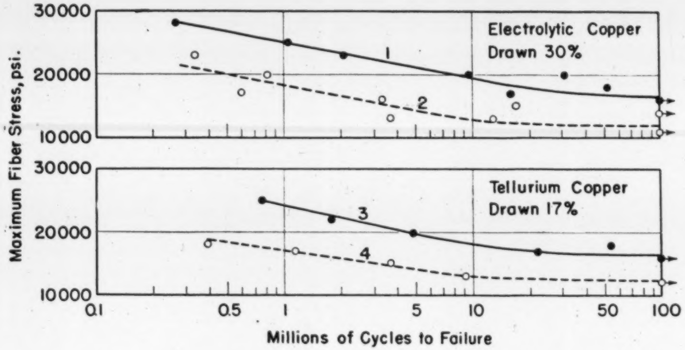


FIG. 1.—*S-N* Curves for Electrolytic Copper and Tellurium Copper.

Polished specimens:
Curves 1 and 3
Notched specimens:
Curve 2, 0.0015-in. notch radius
Curve 4, 0.002-in. notch radius

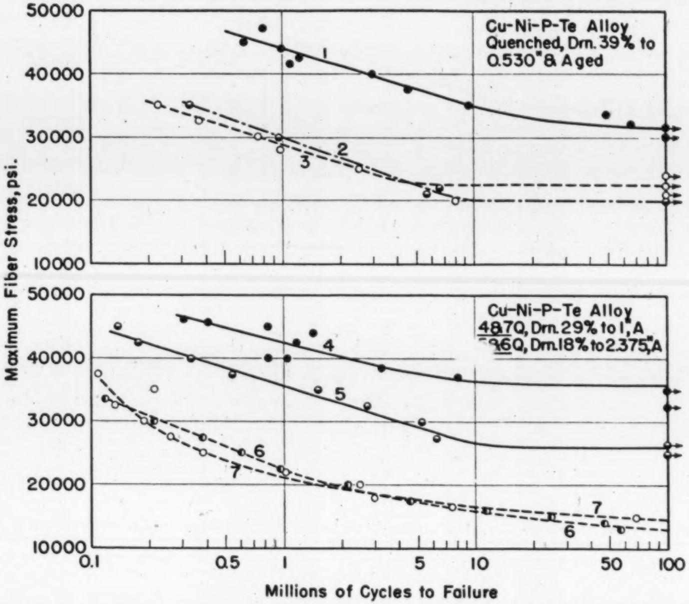


FIG. 2.—*S-N* Curves for Copper-Nickel Phosphorus-Tellurium Alloy.

Polished specimens:
Curves 1, 4, and 5
Notched specimens:
Curve 2, 0.0006-in notch radius
Curve 3, 0.015-in. notch radius
Curve 6, 0.0003-in. notch radius
Curve 7, 0.0003-in. notch radius

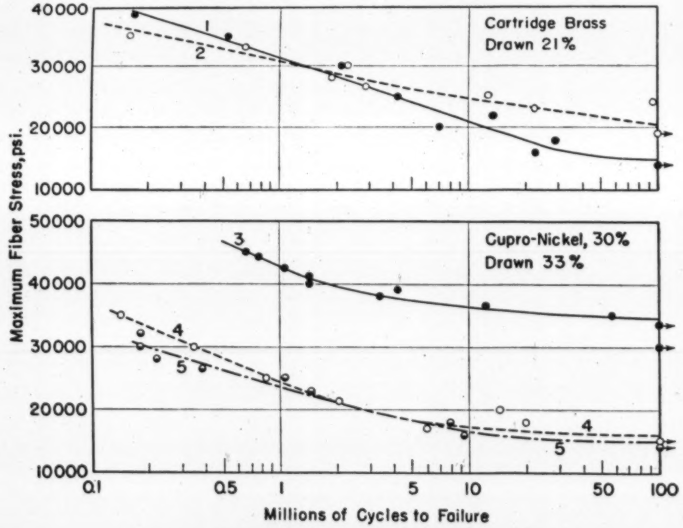


FIG. 3.—*S-N* Curves for Cartridge Brass and Cupro-Nickel, 30 per cent.

Polished specimens:
    Curves 1 and 3
Notched specimens:
    Curve 2, 0.0025-in. notch radius
    Curve 4, 0.015-in. notch radius
    Curve 5, 0.0006-in. notch radius

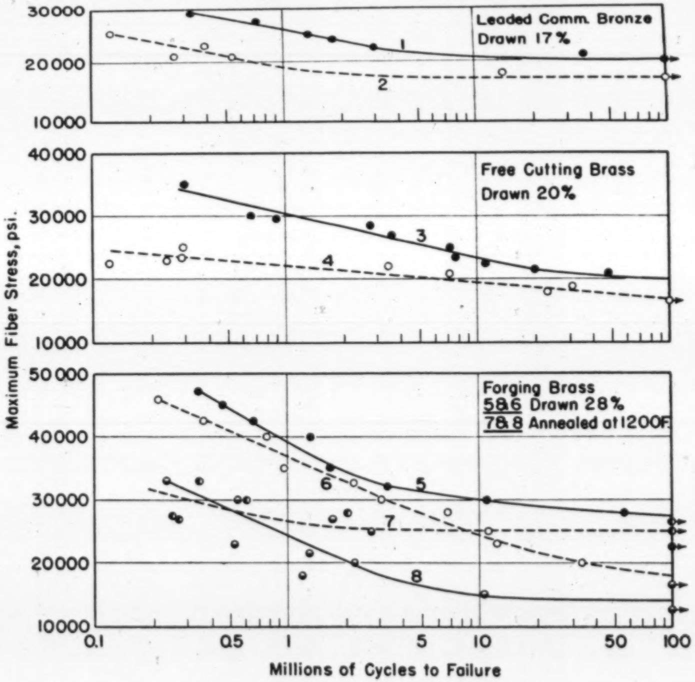


FIG. 4.—*S-N* Curves for Leaded Commercial Bronze, Free Cutting Brass and Forging Brass.

Polished specimens:
    Curves 1, 3, 5, and 8
Notched specimens:
    Curve 2, 0.0015-in. notch radius
    Curve 4, 0.002-in. notch radius
    Curve 6, 0.004-in. notch radius
    Curve 7, 0.005-in. notch radius

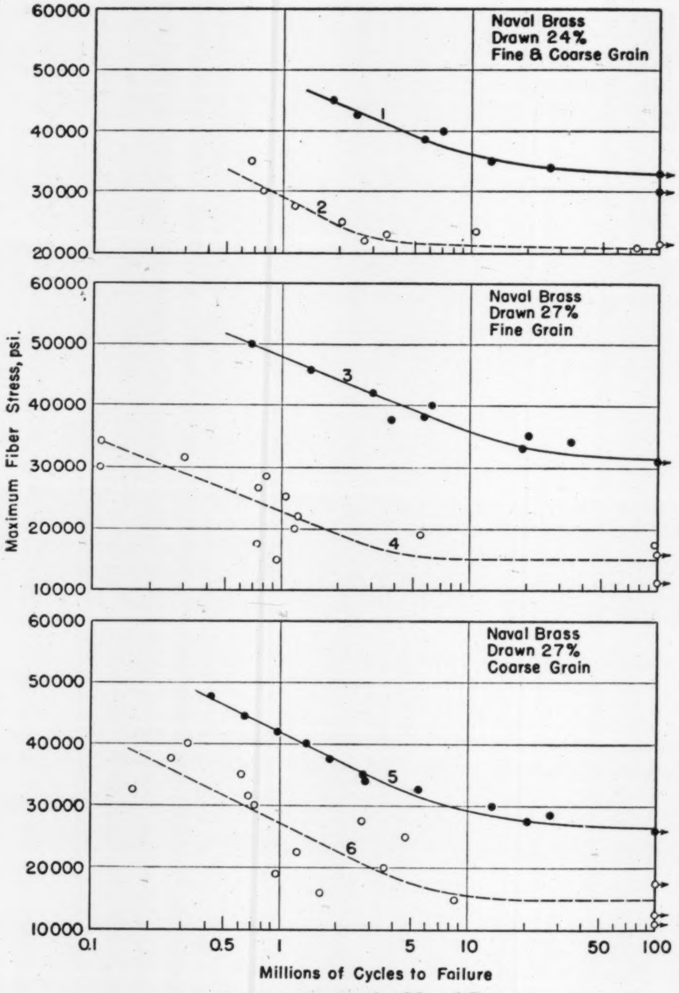


FIG. 5.—*S-N* Curves for Naval Brass.
Polished specimens:
Curves 1, 2, 3, and 5
Notched specimens:
Curves 4 and 6, 0.0006-in. notch radius

fatigue properties. For the drawn materials, the final reduction and the approximate grain size which was produced by the last anneal preceding the final reduction are listed in Table I. The results of tension tests in this table also serve to define further the characteristics of the materials which were tested.

TEST EQUIPMENT AND PROCEDURE

The fatigue tests were made in two Krouse high-speed fatigue testing machines of the rotating-beam type, and a sufficient number of tests was run on each lot of material to establish its *S-N* curve up to 100,000,000 cycles. The test specimen is 4 in. long with a diameter of 0.500 in. at the ends and a minimum reduced section of 0.300-in. diameter at the middle. The specimen is part of a rotating cantilever beam which is loaded by a calibrated, weighted lever suspended from a ball bearing on the free end of the beam. In the tests reported here, the specimens were rotated at approximately 8000 rpm.

The specimens were machined from rods processed to the sizes shown in Table I. The reduced section of the polished or unnotched specimens was produced by machining with a 3-in. radius so that the minimum diameter, 0.300 in., was at the center of the specimen. All tool marks were completely removed by hand polishing with successively finer emery papers. The final longitudinal polishing operation with grade 0000 paper provided a smooth, lustrous surface. The notched specimen was prepared by machining a reduced section of 0.350-in. diameter for a length of 1½ in. in the central portion of the specimen, and thereafter cutting a 60-deg. V notch of 0.025-in. depth in the middle with a tungsten carbide lathe tool. In the earlier part of the work a single tool was used to generate the notch, while later one tool was used to machine the notch to a depth of 0.020 in., and a finishing tool was used to turn to the final depth of 0.025 in. This procedure was adopted to minimize wear of the notching tool so that the notch radius could be duplicated more closely from specimen to specimen. The notch radius for any given group of specimens was determined by sectioning samples and measuring enlarged images of the notches on the viewing screen of a metallographic microscope.

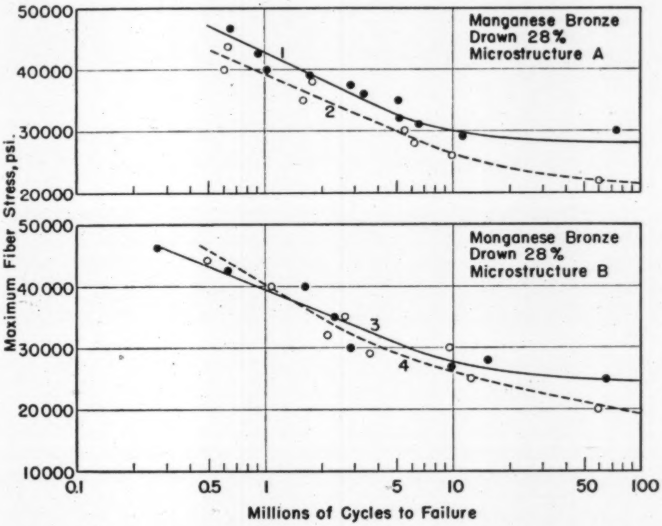


FIG. 6.—*S-N* Curves for Manganese Bronze.

Polished specimens:
Curves 1 and 3
Notched specimens:
Curve 2, 0.0035-in. notch radius
Curve 4, 0.0025-in. notch radius

DISCUSSION

Complete *S-N* curves for all the materials as determined in the fatigue tests are shown in Figs. 1 to 10, and the fatigue strengths at 1,000,000 and 100,000,000 cycles from these *S-N* curves

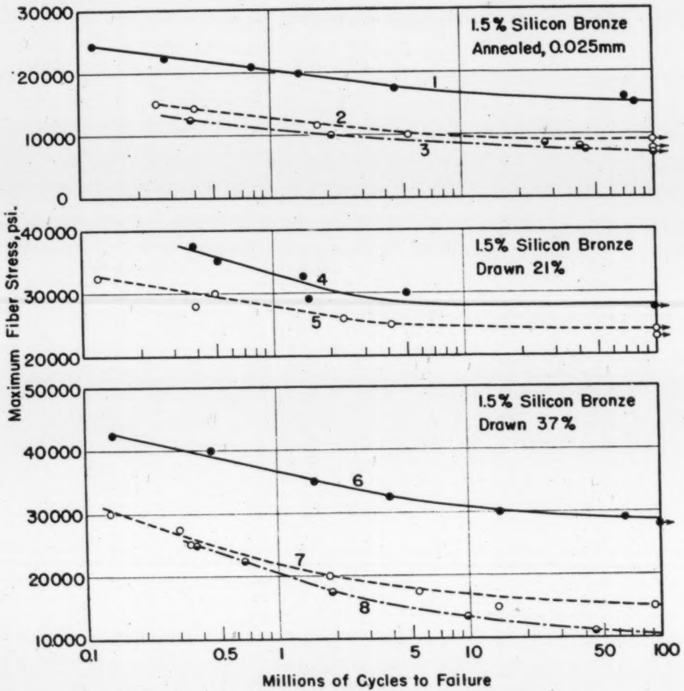


FIG. 7.—*S-N* Curves for 1.5 per cent Silicon Bronze.

Polished specimens:
  Curves 1, 4, and 6
Notched specimens:
  Curves 2 and 7, 0.015-in. notch radius
  Curves 3 and 8, 0.001-in. notch radius
  Curve 5, 0.0015-in. notch radius

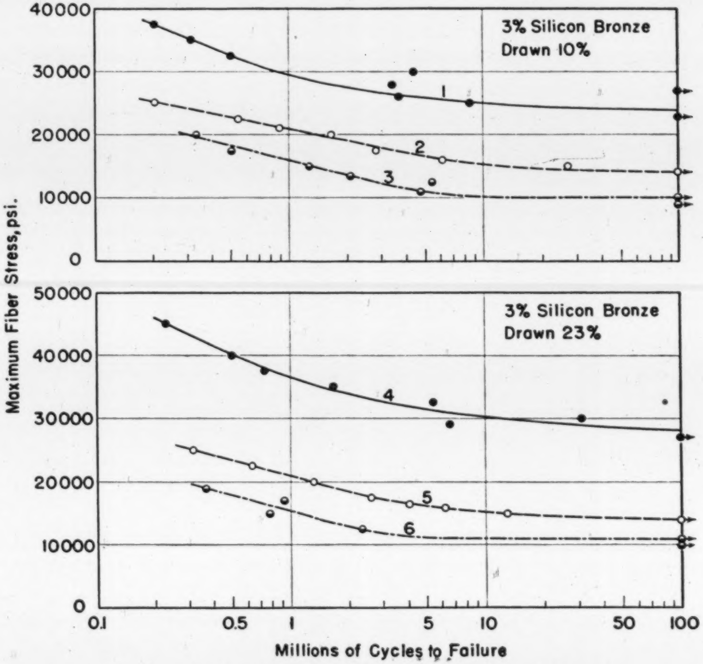


FIG. 8.—*S-N* Curves for 3.0 per cent Silicon Bronze.

Polished specimens:
  Curves 1 and 4
Notched specimens:
  Curves 2 and 5, 0.015-in. notch radius
  Curves 3 and 6, 0.001-in. notch radius

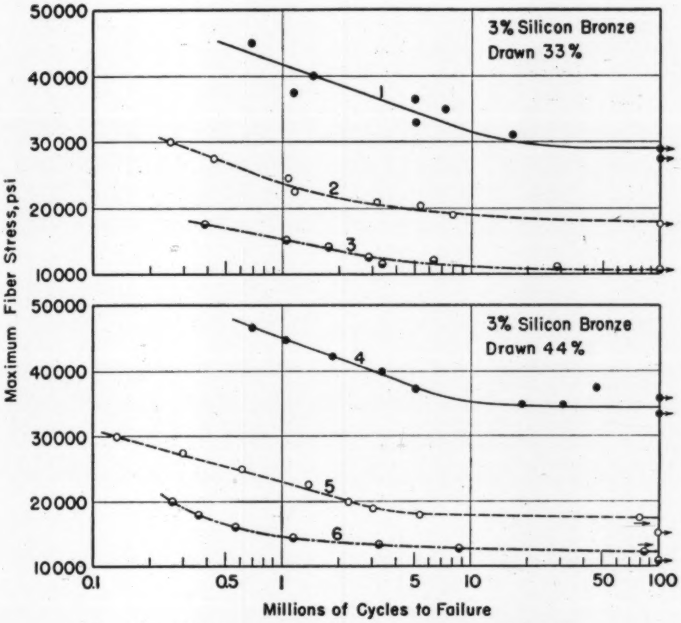


FIG. 9.—*S-N* Curves for 3.0 per cent Silicon Bronze.

Polished specimens:
Curves 1 and 4
Notched specimens:
Curves 2 and 5, 0.015-in. notch radius
Curves 3 and 6, 0.001-in. notch radius

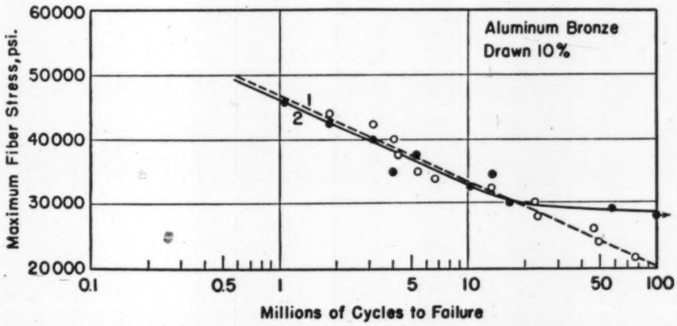


FIG. 10.—*S-N* Curves for Aluminum Bronze.

Polished specimens:
Curve 2
Notched specimens:
Curve 1, 0.0015-in. notch radius

are given in Table I. The ratios of fatigue strength for polished specimens at 100,000,000 cycles to tensile strength are also listed. The notches in the notched specimens are defined in the table by values of notch radii and by the degree of work-hardening of the metal immediately adjacent to the notch, as determined by microscopic examination. The fatigue data were used to compute a fatigue strength reduction or notch-sensitivity factor for the various notched

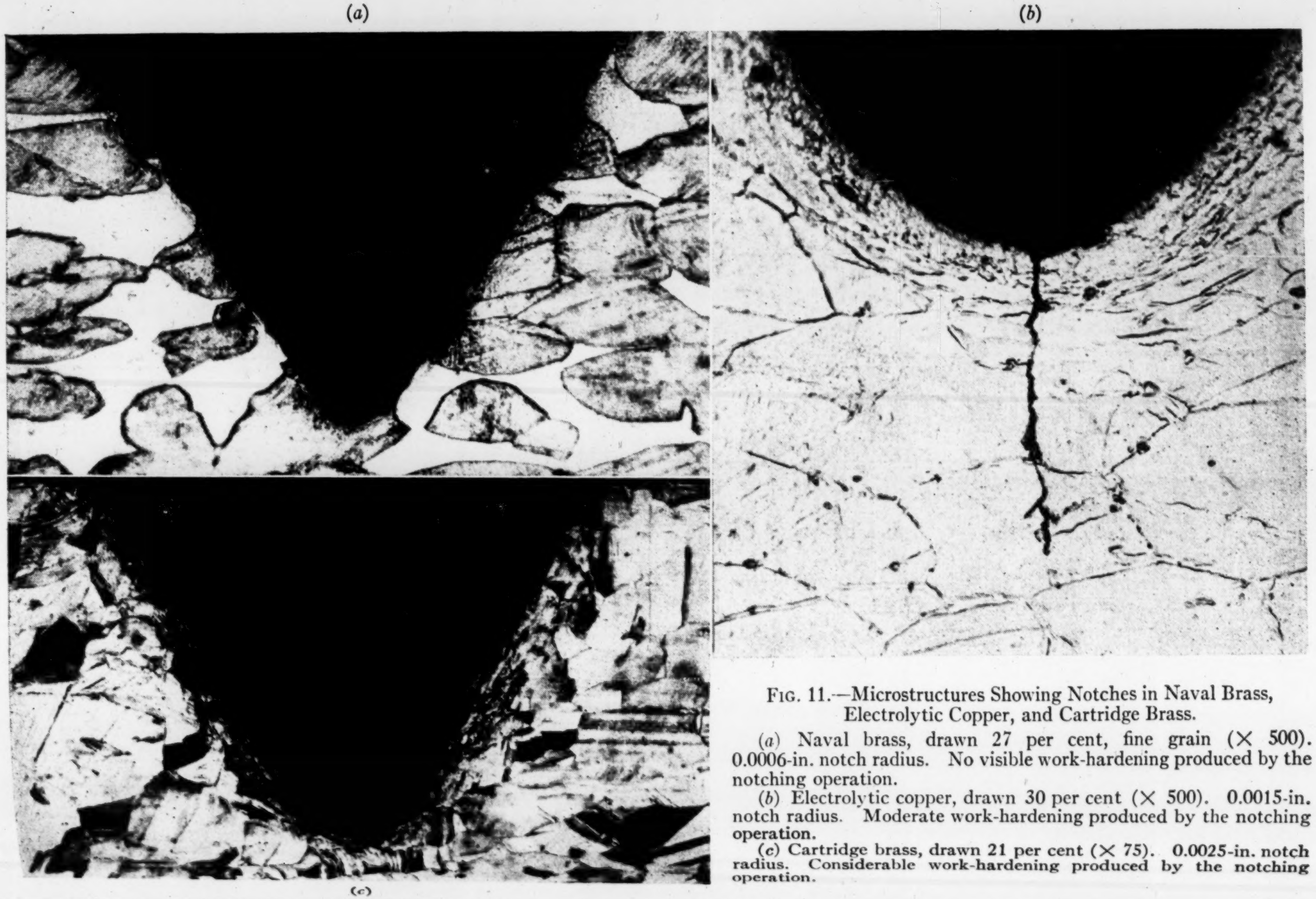


FIG. 11.—Microstructures Showing Notches in Naval Brass, Electrolytic Copper, and Cartridge Brass.

(*a*) Naval brass, drawn 27 per cent, fine grain (× 500). 0.0006-in. notch radius. No visible work-hardening produced by the notching operation.

(*b*) Electrolytic copper, drawn 30 per cent (× 500). 0.0015-in. notch radius. Moderate work-hardening produced by the notching operation.

(*c*) Cartridge brass, drawn 21 per cent (× 75). 0.0025-in. notch radius. Considerable work-hardening produced by the notching operation.

materials. This factor is the ratio of fatigue strength of polished specimens at 100,000,000 cycles to fatigue strength of notched specimens at 100,000,000 cycles.

It is apparent that some of the *S-N* curves tend to level out and indicate a true endurance limit within 100,000,000 cycles while others still show a definite slope at 100,000,000 cycles. There appears to be no clear distinction between the materials of the two groups and the difference may be more apparent than real. It probably emphasizes that a value for fatigue strength should always be accompanied by an indication of the corresponding life or number of cycles to failure.

The materials were tested in notched conditions in order to provide an indication of notch sensitivity by means of the fatigue strength reduction factor. The notches of different lots varied in sharpness, that is, in notch radius, and there was also appreciable variation in the degree of work-hardening of the metal adjacent to the notch. The photomicrographs in Fig. 11 illustrate the range of work-hardening adjacent to notches of the materials tested, that is, (*a*) no visible distortion (Fig. 11(*a*)); (*b*) moderate distortion (Fig. 11(*b*)); (*c*) considerable distortion (Fig. 11(*c*)). This was particularly true in the earlier part of the work when a single notching tool was used. Therefore the fatigue strength reduction factors listed in the table cannot be readily used as a common measure of relative notch sensitivity because all of them do not refer to a common notch. When the notches were more precisely made, regardless of actual root radius for a given group, the amount of scatter of points on the *S-N* curves for notched specimens was scarcely greater than the scatter for polished specimens, except in the case of naval brass. For this alloy the notched specimens produced a scatter of points which was much greater than in the average case. It is unlikely that any possible variation in notch radius or extent of cold working produced by the notching operation could account for the abnormally large scatter in this instance.

Lowering of fatigue strength, which is normally effected by notching, was realized in nearly all of the tests reported here. With regard to the effect of notch radius, the present results indicate an increasing effect as notch radius decreases; for example, notches of approximately 0.001-in. root radius produced greater decrease of fatigue strength than did the notches of about 0.015-in. radius. This is consistent with the generally expected relationship between stress concentration and notch radius, that is, the sharper the notch, the greater the stress concentration.

The factor of work-hardening the metal in the notching operation is a very important one and may easily exceed the effect of rather large variations in notch radius. The greatest decrease in fatigue strength is produced when the structure of the remaining metal is undisturbed during the notching operation. If the notch is machined improperly, the metal immediately adjacent to the notch may be distorted and strain-hardened. In so far as endurance is concerned, this condition produces a strengthening effect which may be attributed in part to the naturally higher fatigue strength of most cold-worked metals as compared with the same metals either less severely worked or annealed, and in part to the fact that this localized working of the metal produces a state of compression in the surface layers of the metal somewhat similar to that effected by shot-peening. It is well known that such a state of compression operates to increase fatigue strength of metals. An example of the marked effect of working the metal at the notch occurs in the case of cartridge brass, where the fatigue strength of the notched specimens is 20,500 psi. at 100,000,000 cycles and the corresponding value obtained on unnotched, polished specimens was only 15,000 psi. The metal adjacent to the notches had been very considerably work hardened by the notching tool, as shown in Fig. 11(*c*). The detrimental effect of a notch thus may be minimized or completely eliminated by this work-hardening, and on this basis it is conceivable that notches, if unavoidable, might best be made in such a way that a significant degree of localized work-hardening is imparted to the metal.

It is apparent from the results shown in Table I that the fatigue properties of the copper-base alloys included in this investigation differ widely with composition and temper. The relative merits of the various compositions cannot be readily evaluated even for unnotched specimens because of the differences in temper. Considering all of the materials, the results show that the fatigue strengths at 100,000,000 cycles for polished specimens range from 14,000 to 36,000 psi. By excluding materials in which there was considerable work-hardening adjacent to the notches and without taking notch radius into account, the lowest fatigue strength at 100,000,000 cycles for notched specimens of these materials is 7,000 psi. and the highest is 22,500 psi.

Referring to polished specimens, the minimum value of the ratio of fatigue strength to tensile strength for all of the materials tested is 0.26 and the maximum is 0.52. There is no simple correlation of fatigue and tensile strengths. High fatigue strengths are sometimes associated with low ratios of fatigue strength to tensile strength and low fatigue strengths are frequently associated with high ratios (see Table I). Values of similar ratios for notched specimens were not computed because the values of tensile strength as determined on unnotched and notched specimens are not equal, and neither one offers an adequate basis for analysis of fatigue characteristics of notched specimens.

TABLE II.—CORRELATION OF FATIGUE STRENGTH REDUCTION FACTOR WITH NOTCH RADIUS FOR SEVERAL MATERIALS.

Material	Temper	Notch Radius, in.	Fatigue Strength Reduction Factor
Cu-Ni-P-Te alloy (0.530-in. diameter)	Quenched, drawn 39% and aged	3	1
		0.015	1.4
		0.0006	1.6
Cupro-nickel, 30 per cent	Drawn 33%	3	1
		0.015	2.2
		0.0006	2.3
1.5 per cent silicon bronze	Annealed, 0.025-mm. grain size	3	1
		0.015	1.7
		0.001	2.1
1.5 per cent silicon bronze	Drawn 37%	3	1
		0.015	1.9
		0.001	2.7
3 per cent silicon bronze	Drawn 10%	3	1
		0.015	1.7
		0.001	2.4
3 per cent silicon bronze	Drawn 23%	3	1
		0.015	2.0
		0.001	2.5
3 per cent silicon bronze	Drawn 33%	3	1
		0.015	1.6
		0.001	2.8
3 per cent silicon bronze	Drawn 44%	3	1
		0.015	1.9
		0.001	2.8

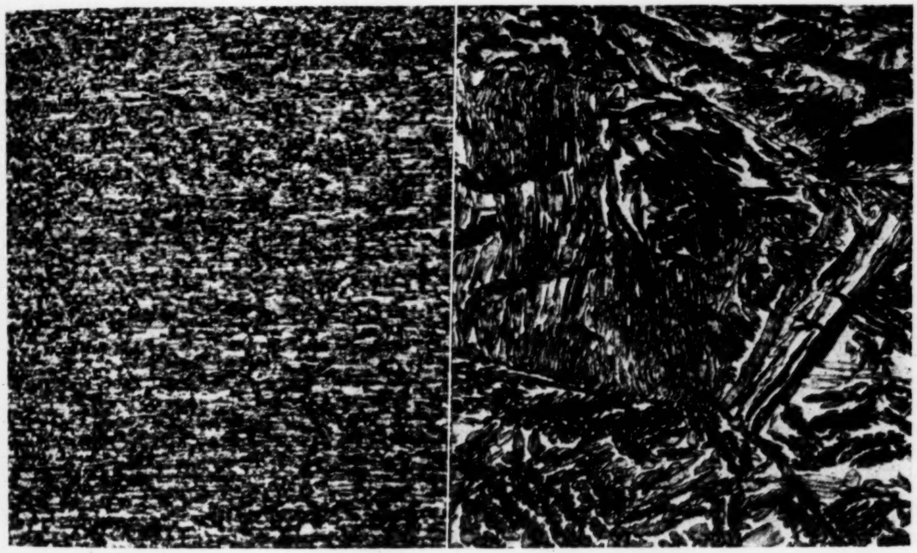
The fatigue strength reduction factors for all groups of notched specimens, except those where there was considerable work-hardening adjacent to the notch, range from 1.2 to 2.8. Thus the fatigue strengths of most of the copper-base materials tested in this investigation are reduced by notch sensitivity to values which range from about 35 to 83 per cent of the fatigue strengths for polished specimens. These values were calculated without regard to the individual degrees of notch sharpness, which ranged from 0.0003-in. root radius to 0.015-in. root radius.

While the variety of notched conditions prevent a truly quantitative comparison of the test materials with regard to notch sensitivity, it is to be seen in Table I that the lead-bearing brasses and tellurium copper have generally lower fatigue strength reduction factors, indicating lower notch sensitivity, than the remainder of the materials.

The effect of increasing notch sharpness on notch sensitivity for each of eight materials is evaluated in Table II. Polished specimens are arbitrarily designated here as representing a 3-in. notch radius with a fatigue strength reduction factor of unity. There is an increase in fatigue strength reduction factor as notch radius decreases in each case, and presumably the extreme effect would be that produced by a fine crack.

Naval Brass:

In evaluating the fatigue properties of naval brass, Anderson and Smith[2] reported distinct differences between materials processed (*a*) by hot rolling, and (*b*) by hot extrusion before the final cold-drawing operation. By inference, this may be attributed to some structural difference. A brief study has been made in the present work which clarifies the effect of microstructure on fatigue characteristics of naval brass.



(*a*) Fine grain (*b*) Coarse grain

FIG. 12.—Microstructures of Naval Brass, Drawn 24 per cent (× 50).

Two lots of naval brass, having almost identical compositions and tensile strengths, were produced by the following sequence of operations: extruded to 0.695 in. under conditions which produced a fine-grained structure, cold-drawn to 0.575 in., annealed as indicated below, and cold-drawn 24 per cent to 0.500 in. The first lot was given a light commercial anneal at 900 F. on 0.575 in., and the second lot was annealed at 1450 F. The microstructure after the anneal at the lower temperature was essentially the same as that for the extruded condition, namely, a fine-grained structure of equiaxed alpha grains uniformly dispersed in the matrix of the beta phase. The anneal at 1450 F. produced a structure of acicular grains of alpha precipitated within very coarse grains of beta. The microstructures of the two lots after the final reduction are shown in Fig. 12. The fatigue strength of the first lot, which has the fine-grained structure, is 33,000 psi. at 100,000,000 cycles (polished specimens), while that of the second lot, which has the coarse-grained structure, is only 21,000 psi. Inasmuch as the structural difference is the only apparent one, it is reasonable to consider that it accounts for this difference in fatigue strength.

TABLE III.—EFFECT OF MICROSTRUCTURE ON FATIGUE STRENGTH OF NAVAL BRASS.

Temper, per cent reduction	General Designation of Microstructure	Relative Microstructure	Fatigue Strength at 100,000,000 Cycles, psi. (Polished Specimens)
24..........	Fine grain	Very fine	33 000
27..........	Fine grain	Fine	31 500
27..........	Coarse grain	Coarse	26 500
24..........	Coarse grain	Very coarse	21 000

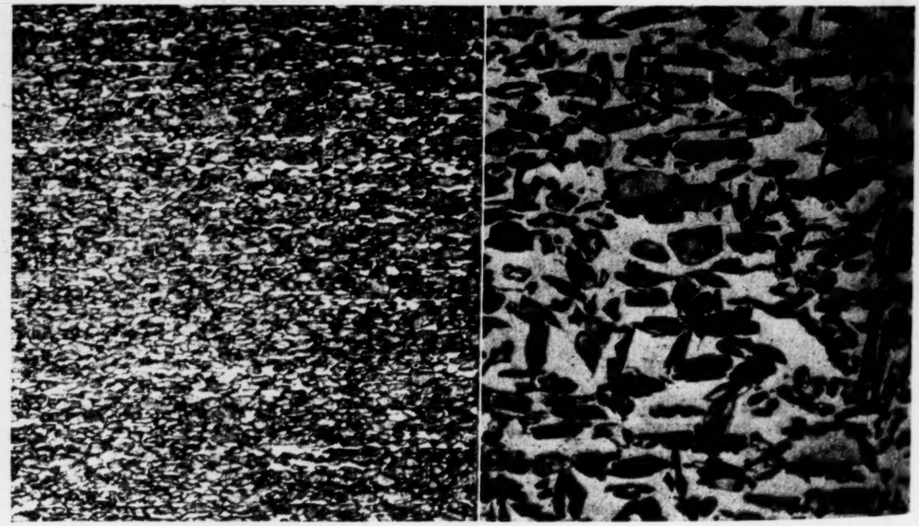

(*a*) Fine grain (*b*) Coarse grain

FIG. 13.—Microstructures of Naval Brass, Drawn 27 per cent (× 50).

Further evidence of the same kind is derived from the polished specimens of the lots of naval brass as drawn 27 per cent. These lots were processed by extruding, cold-drawing, annealing, and redrawing 27 per cent to the final size. The two structures shown in Fig. 13(*a*) and (*b*), were obtained by annealing at relatively low and high temperatures, respectively, prior to the final reduction. Here there is a lesser difference in microstructure and also in fatigue strength, but again the finer, more uniform structure is the one which produces the better fatigue-resisting properties, 31,500-psi. fatigue strength as compared with 26,500 psi. for the coarser structure. The results of tests on polished specimens of all four lots of the naval brass rod made by the extrusion process show that the structural factor predominates over minor differences in degree of cold-drawing and tensile strength. The values of fatigue strength for polished specimens of the four lots decrease as the microstructure becomes coarser, as shown in Table III. Thus it is seen that variation in structure, rather than manner of processing, can adequately account for a very considerable variation in fatigue strength of this alloy.

Manganese Bronze:

The fatigue strength of manganese bronze was also found to vary with microstructure. Two lots were prepared by annealing extruded and cold-drawn rod at 1200 and 1400 F., respectively, and drawing 28 per cent to produce the two structures, A and B, shown in Fig. 14. Structure A consists of small, residual alpha grains, in a matrix of beta grains which were found by microscopic examination to be only slightly larger than the alpha grains. The boundaries of the beta grains cannot be distinguished in the photomicrograph. Structure B consists of acicular grains of alpha precipitated in a very coarse-grained beta matrix. The coarseness of the latter structure is not evident in the photomicrograph because only a portion of a single beta grain is visible. The values of fatigue strength for polished specimens at 100,000,000 cycles are 28,000 psi. for the finer structure, A, and 24,500 psi. for the coarser structure, B. The effect of structural difference is much less pronounced in the case of the notched specimens. Tensile properties are virtually independent of the structural difference.

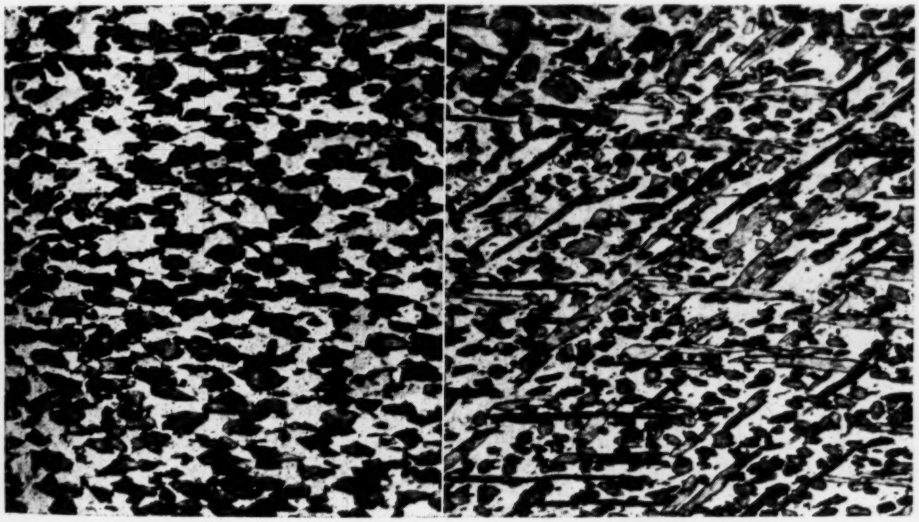

Structure A Structure B

FIG. 14.—Microstructures of Manganese Bronze, Drawn 28 per cent (× 75).

3 per cent Silicon Bronze:

The 3 per cent silicon bronze was investigated in four drawn tempers, namely, as drawn 10, 23, 33, and 44 per cent. Some of the tension and fatigue data for these materials are shown graphically in Fig. 15. The rate of increase of tensile strength with increasing reduction is considerably greater than is the corresponding increase of fatigue strength. The tensile strength increases 64 per cent from 10 per cent reduction to 44 per cent reduction. The corresponding increases in fatigue strength at 100,000,000 cycles are 42 per cent for polished specimens and 25 and 20 per cent for notched specimens with 0.015 and 0.001-in. notch radii, respectively. It is thus apparent that fatigue strength of this alloy increases with degree of cold-working to an extent which depends on whether the surface is polished or notched. Notches offset in part the strengthening effect of greater reductions. The additional gain in fatigue strength effected by heavier reductions diminishes as the notch sharpness increases.

SUMMARY

The fatigue properties of the materials tested in this investigation vary widely with composition and temper. For polished specimens of these materials the fatigue strengths at 100,000,000 cycles range from 14,000 psi. to 36,000 psi. The corresponding values for notched specimens range from 7000 psi. to 22,500 psi., excluding those materials in which considerable work hardening was produced in the notching operation. The results show that such work hardening of the metal adjacent to the notch can offset at least in part the normal reduction of fatigue strength due to notching. The ratios of fatigue strength at 100,000,000 cycles for polished specimens to tensile strength for all the materials range from 0.26 to 0.52. The fatigue strength reduction factors for the various materials and for the different degrees of notch sharpness range from 1.2 to 2.8. Notch sensitivity, as indicated by fatigue strength reduction factor, increases with degree of notch sharpness.

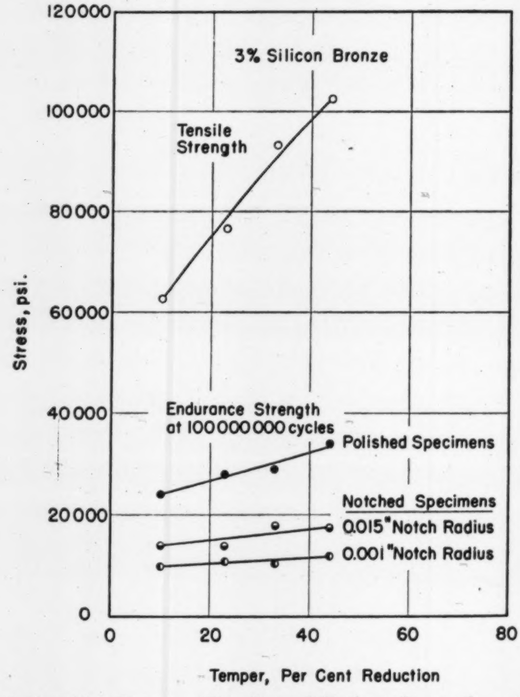


FIG. 15.—Relationships Between Tensile and Fatigue Strengths and Temper of 3 per cent Silicon Bronze.

The fatigue properties of naval brass and manganese bronze are particularly dependent on their microstructures. The fatigue strength of 3 per cent silicon bronze increases with increasing degree of cold working.

Acknowledgments:

The authors acknowledge the interest of D. K. Crampton, Director of Research of the Chase Brass and Copper Co., in this fatigue testing program and the assistance of those who participated in various phases of the work. Permission to publish the results was given by the Chase Brass and Copper Co.

DISCUSSION

MR. R. E. PETERSON.[1]—The statement "notch sensitivity increases with notch sharpness" calls for some comment. Terminology is not well established in the fatigue field and what is probably intended is that the "*fatigue strength reduction factor* increases with notch sharpness." This latter term is defined

$$K_f = \frac{\text{Fatigue strength without notch}}{\text{Fatigue strength with notch}}$$

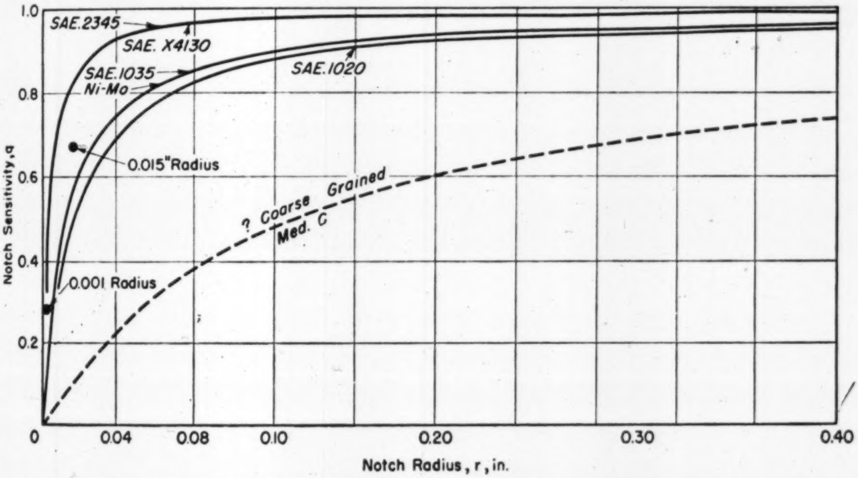


FIG. 16.—Average Notch Sensitivity Curves (Notch Depth-Notch Radius Ratio < 3).

Notch sensitivity is defined:

$$q = \frac{K_f - 1}{K_t - 1}$$

where K_t = stress concentration factor (theoretical).

If we use Neuber's values for K_t, we obtain 2.2 for 0.015-in. notch radius and 6.6 for 0.001-in. radius.[2] The calculated q values average 0.67 for the 0.015-in. radius and 0.28 for the 0.001-in. radius.

These results are shown superposed on the accompanying Fig. 16, which was published in the March, 1945, issue of the ASTM BULLETIN.[3] It seems, then, that in general, notch sensitivity *decreases* with notch sharpness, especially as the radius becomes quite small.

As mentioned at the outset, we have not done a good job with regard to definitions and terminology, but it is expected that Committee E-9 on Fatigue will clarify this situation in a Manual which is now in preparation.

The present paper by Burghoff and Blank is an excellent one, offering a wealth of carefully obtained source data. All of the precautions required for good testing, as now being discussed in Committee E-9 in connection with the above mentioned Manual, have been taken. It is a paper that will be of permanent value.

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[1] Manager, Mechanics Div., Westinghouse Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa., Chairman of Committee E-9 on Fatigue.

[2] The K_t values given have also been modified in accordance with the shear energy theory, but this is not a large effect and has no bearing on the conclusions.

[3] R. E. Peterson, "Relation Between Life Testing and Conventional Tests of Materials," ASTM BULLETIN, No. 133, March, 1945, p. 14.

MR. A. I. BLANK (*author's closure*).—We are grateful to Mr. Peterson for his comments on notch sensitivity and agree with him that fatigue strength reduction factors alone do not indicate the variation of notch sensitivity with notch sharpness. No attempt was made in this investigation to determine true stresses in the notched specimens. The notch sensitivities of the various materials were compared only on the basis of their fatigue strength reduction factors as defined by the ratio of the fatigue strength of unnotched specimens to the fatigue strength of notched specimens at 100 million cycles. We recognize that the criterion of notch sensitivity which Mr. Peterson suggested provides a more precise basis of analysis.

THE FATIGUE CHARACTERISTICS OF COPPER-NICKEL-ZINC AND PHOSPHOR BRONZE STRIP IN BENDING UNDER CONDITIONS OF UNSYMMETRICAL LOADING*

By G. R. Gohn[1] and W. C. Ellis[1]

Synopsis

This paper describes the results of a series of fatigue tests in bending on two non-ferrous materials in strip form under conditions of unsymmetrical loading. A description is given of the machine and test methods developed for making such tests on thin strip metal. Included in the paper are *S-N* curves as well as other curves showing the effect of mean stress on the range of stress in unsymmetrical repeated bending.

Fatigue data in bending under conditions of completely reversed stress have been published in previous papers presented before the Society (**1**, **2**, **3**,)[2] and elsewhere (**4**) for most of the common non-ferrous metals in strip form. Such data have proved very useful in the design of equipment and apparatus involving the use of small flat springs. In some cases the springs are subjected to complete reversal of stress during operation. In most cases, however, such springs are operated under conditions of unsymmetrical loading in which the initial stress is either zero or a small value corresponding to an initial contact pressure. Subsequently, these springs are operated to some point of greater deflection or higher contact pressure. The fatigue characteristics under these conditions (that is, for various mean stresses) are, therefore, of great engineering importance.

Since the determination of the fatigue strength values for a comprehensive range of mean stresses becomes a lengthy experimental task, it becomes desirable to establish a relationship between the fatigue strength for complete stress reversal and any practical mean stress. Experimental data have been published for many materials, both brittle and ductile, in unsymmetrical, torsional or tension and compression loading, and general relationships for such loading conditions (usually adequate for design considerations) have been developed. Because such data have not been published for unsymmetrical bending stresses, an investigation was made of the fatigue characteristics of two widely used spring materials, alloy No. 4[3] copper-nickel-zinc (18 per cent nickel silver) strip in extra hard temper and grade C[4] phosphor bronze (nominal 8 per cent tin) strip in spring temper under these loading conditions. This

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 721.

[3] Tentative Specifications for Copper Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 - 46a T), 1946 Book of A.S.T.M. Standards, Part I-B, p. 402.

[4] Standard Specifications for Phosphor Bronze Sheet and Strip (B 103 - 46), 1946 Book of A.S.T.M. Standards, Part I-B p. 74.

paper discusses the results of those tests and suggests a relationship between the fatigue strength for complete reversal of stress (symmetrical loading) and the fatigue strength under conditions of unsymmetrical loading.

DISCUSSION OF PREVIOUS WORK

The general problem of developing a relationship between range of stress and mean stress has had the attention of engineers for many years and in the early part of the century J. Goodman (5) and J. B. Johnson (6) published independently equivalent analyses. The original formula (generally known as the Johnson-Goodman formula) has since been modified by replacing the term, one third of the static ultimate tensile strength, with the experimentally determined endurance limit for complete reversal of stress. The resulting modified formula is:

$$S_a = S_r \left(1 - \frac{S_m}{S_u}\right) \quad \ldots\ldots\ldots (1)$$

where:

S_a = the alternating stress component of a range of stress in pounds per square inch, or

$$S_a = \frac{S_{\max} - S_{\min}}{2}$$

$S_{\max}$ = the maximum (numerical) stress of a range of stress in pounds per square inch.

$S_{\min}$ = the minimum (numerical) stress of a range of stress in pounds per square inch.

S_r = the endurance limit in pounds per square inch for the material in question under conditions of complete stress reversal. (In this paper the term S_r is used to denote the fatigue strength of "as-rolled", cross-milled specimens of strip material for 10^8 cycles of completely reversed stress.)

S_m = the mean stress component of a range of stress in pounds per square inch or

$$S_m = \frac{S_{\max} + S_{\min}}{2}$$

S_u = the static ultimate tensile strength in pounds per square inch.

Numerous other formulas proposed for relating fatigue characteristics to a range of stress have been critically discussed by H. F. Moore and J. B. Kommers (7, 8).

An excellent analysis of published fatigue data obtained for unsymmetrical loading tests made prior to 1942 has been made by J. O. Smith (9). Included in the analysis are data on both ductile and brittle materials for tests in torsion and in axial tension and compression. For ductile materials in torsion, the alternating stress was constant for various mean stresses, provided the maximum stress did not exceed the yield point. For notch-free ductile specimens in which the mean stress was axial tension, the alternating stress could be computed from Equation 1. However, when the mean stress was axial compression, the alternating stress was either constant and equal to or greater than the endurance limit for completely reversed stress.

Orowan (10), on theoretical grounds, has shown that the safe range of stress is independent of the mean stress of the cycle so long as the maximum stress does not exceed the elastic limit. This conclusion arises from a theory of fatigue failure which postulates that plastic deformation is not homogeneous.

MATERIALS AND METHOD OF TEST

The materials tested were commercial lots of 0.0201 in. thick, extra hard

TABLE I.—CHEMICAL ANALYSIS.

Composition, per cent	Alloy No. 4 Copper-Nickel-Zinc	Grade C Phosphor Bronze	
		0.0201-in stock	0.0320-in. stock
Copper	55.09	92.04	92.01
Lead	...	0.01	0.01
Iron	0.04	0.02	0.01
Zinc	26.31	0.16	0.17
Tin	...	7.68	7.68
Phosphorus	...	0.066	0.057
Antimony	..	trace	nil
Nickel	18.35	...	...
Manganese	0.16	...	...

temper alloy No. 4 copper-nickel-zinc strip and 0.0320 in. thick, spring temper, grade C phosphor bronze strip. In the case of the latter, the *S-N* diagrams for complete stress reversal were determined on 0.0201-in. thick material. Because of the large deflections involved, this thickness of phosphor bronze could not be tested in unsymmetrical loading. Sufficient tests were, however, run on the 0.0320-in. phosphor bronze to determine that the fatigue strength at 10^8 cycles of completely reversed stress was 26,000 psi., the same as that of the 0.0201-in. stock. The chemical analyses of the two materials are given in Table I and the mechanical properties in Table II.

The specimens used in these fatigue tests are those described by Greenall and Gohn in a previous paper (**2**). Tests were made on the specimens with the "as-rolled" finish.

A fatigue machine was so designed for these tests that the marked change in length of the specimens for the large deflections, necessary in the unsymmetrical loading tests, could be accommodated. This machine is shown schematically in Fig. 1.

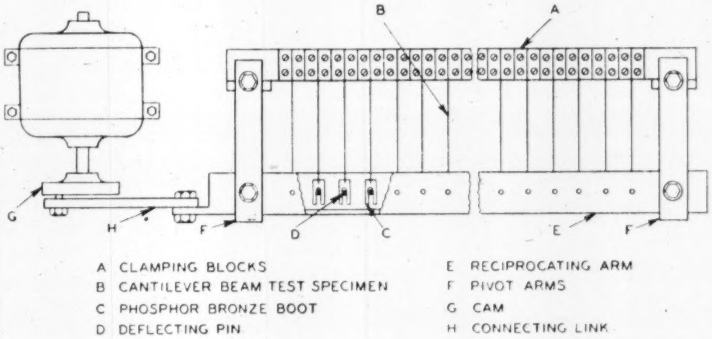

FIG. 1.—Schematic Drawing of Fatigue Testing Machine for Unsymmetrical Loading Tests in Bending

TABLE II.—MECHANICAL PROPERTIES.

Physical Properties	Extra Hard Temper, Alloy No. 4 Copper-Nickel-Zinc	Spring Temper, Grade C Phosphor Bronze
Tensile strength, psi. (S_u)	102 200	112 900
Proportional limit, psi.	48 300	56 100
Yield strength at 0.005 per cent offset, psi.	60 000	66 500
Yield strength at 0.01 per cent offset, psi.	81 600	82 200
Modulus of elasticity, psi.	19 500 000	15 100 000
Elongation in 2 in., per cent	1.0	1.5
Fatigue strength at 10^8 cycles, psi. (S_r)	21 000	26 000
Ratio $\frac{\text{fatigue strength}}{\text{tensile strength}} = \frac{(S_r)}{(S_u)}$	0.205	0.230

It consists essentially of a series of clamping blocks *A* in which one end of the test specimens *B* is mounted. The free end of the test specimen is soldered to a phosphor bronze boot *C* which is deflected by a pin *D* fastened to a motor-driven deflecting arm *E*.

TABLE III.—*S-N* DATA—RANGE OF STRESS *versus* NUMBER OF CYCLES ENDURED.

A. EXTRA HARD TEMPER, ALLOY NO. 4 COPPER-NICKEL-ZINC STRIP

Range of Stress, psi.....	(−)18 500 to (+)18 500[a]	(−)21 600 to (+)21 600	(−)24 700 to (+)24 700	(−)30 900 to (+)30 900	(−)37 000 to (+)37 000	(−)43 200 to (+)43 200
Number of Cycles....	100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→	4 766 000 5 713 000 7 055 000 13 675 000 42 275 000	3 424 000 3 750 000 5 257 000 11 643 000	1 355 000 1 577 000 1 582 000 1 636 000 1 736 000	493 000 577 000 696 000 720 000 745 000	307 000 308 000 319 000 358 000
Range of Stress, psi....	0 to (+)33 300	0 to (+)42 300	0 to (+)52 800	0 to (+)62 200		
Number of Cycles....	100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→	6 000 000 6 150 000 6 900 000 9 100 000 17 200 000	850 000 1 220 000 1 220 000 1 430 000 1 480 000	490 000 490 000 510 000 575 000 575 000		
Range of Stress, psi....	(+)6 500 to (+)39 800	(+)6 500 to (+)48 800	(+)6 500 to (+)59 300	(+)6 500 to (+)68 700		
Number of Cycles....	100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→	2 050 000 4 000 000 4 300 000 4 300 000 5 250 000	860 000 860 000 1 040 000 1 080 000 1 080 000	400 000 437 000 490 000 490 000 570 000		
Range of Stress, psi....	(+)13 000 to (+)46 300	(+)13 000 to (+)55 300	(+)13 000 to (+)65 800	(+)13 000 to (+)75 200		
Number of Cycles....	100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→	3 750 000 4 420 000 5 100 000 5 500 000 10 400 000	735 000 1 040 000 1 080 000 1 280 000 1 280 000	400 000 432 000 480 000 515 000 515 000		
Range of Stress, psi....	(+)19 500 to (+)52 800	(+)19 500 to (+)61 800	(+)19 500 to (+)72 300	(+)19 500 to (+)81 700		
Number of Cycles....	100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→	2 020 000 3 000 000 4 150 000 5 250 000 39 500 000	1 020 000 1 020 000 1 280 000 1 470 000 2 930 000	405 000 405 000 510 000 510 000 510 000		

B. SPRING TEMPER, GRADE C PHOSPHOR BRONZE STRIP

Range of Stress, psi.....	(−)19 900 to (+)19 900	(−)22 100 to (+)22 100	(−)24 300 to (+)24 300	(−)26 500 to (+)26 500	(−)30 900 to (+)30 900
Number of Cycles....	100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→	100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→	100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→	28 268 000 30 854 000 46 765 000 57 854 000→ 57 854 000→	1 600 000 1 601 000 1 618 000 3 081 000 4 518 000
Range of Stress, psi....	0 to (+)48 200	0 to (+)60 500	0 to (+)72 500		
Number of Cycles....	100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→	685 000 920 000 5 300 000 19 400 000 48 500 000	192 000 192 000 213 000 237 000 293 000		
Range of Stress, psi....	(+)7 400 to (+)55 600	(+)7 400 to (+)67 900	(+)7 400 to (+)79 900		
Number of Cycles....	100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→	3 250 000 15 300 000 46 500 000 48 000 000 51 000 000	192 000 295 000 265 000 270 000 287 000		
Range of Stress, psi....	(+)14 900 to (+)63 100	(+)14 900 to (+)75 400	(+)14 900 to (+)87 400		
Number of Cycles....	100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→	355 000 680 000 680 000 4 900 000 42 500 000	215 000 225 000 225 000 225 000 255 000		
Range of Stress, psi....	(+)22 400 to (+)70 600	(+)22 400 to (+)82 900	(+)22 400 to (+)94 900		
Number of Cycles....	100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→ 100 000 000→	3 850 000 4 950 000 14 300 000 26 500 000 46 800 000	190 000 202 000 210 000 242 000 268 000		

[a] → Indicates specimen unbroken after number of cycles indicated.

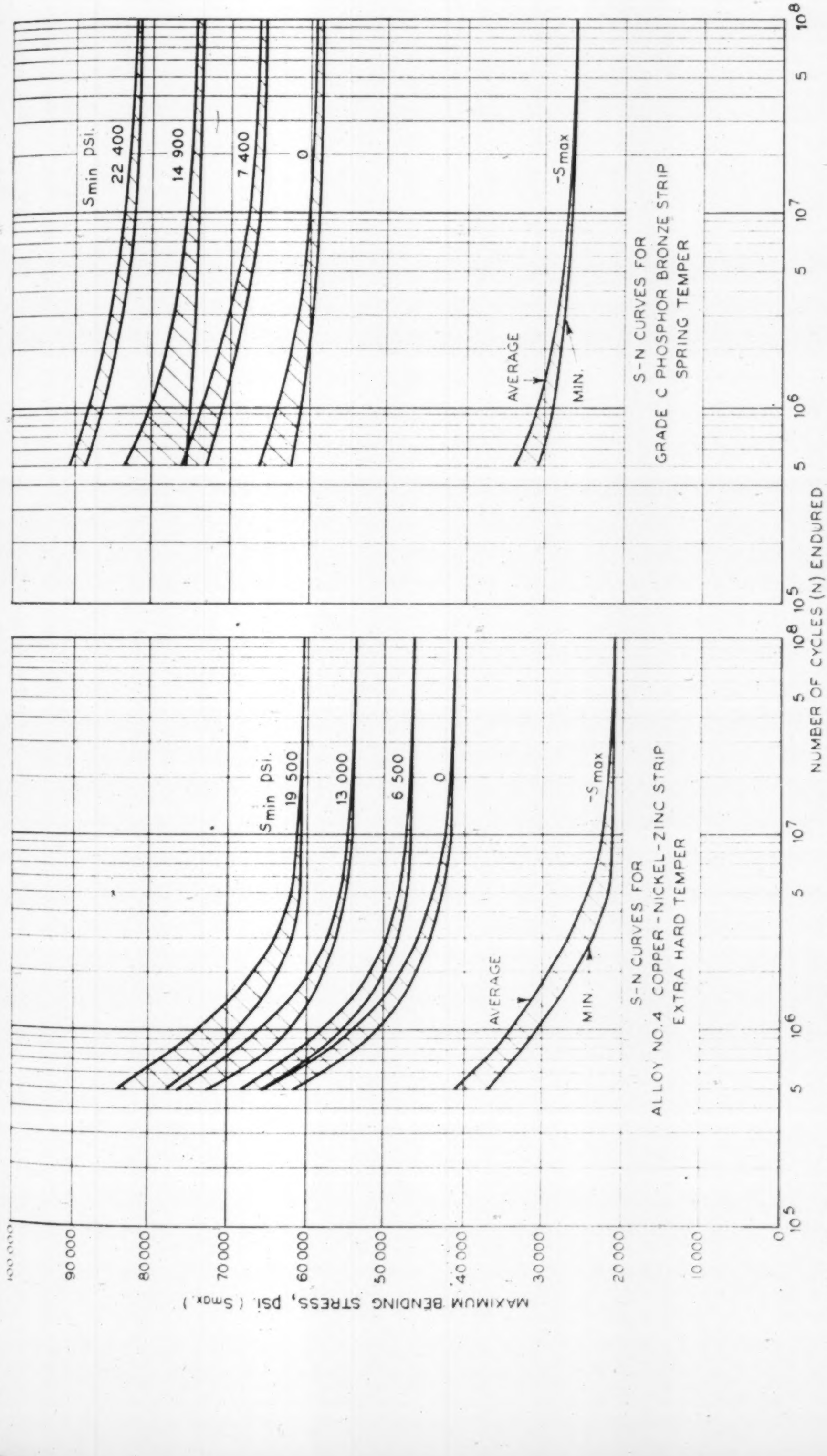

FIG. 2.—S-N Curves Showing Relation Between Maximum Bending Stress and Number of Cycles Endured at Constant Minimum Stress and for Complete Reversal of Stress.

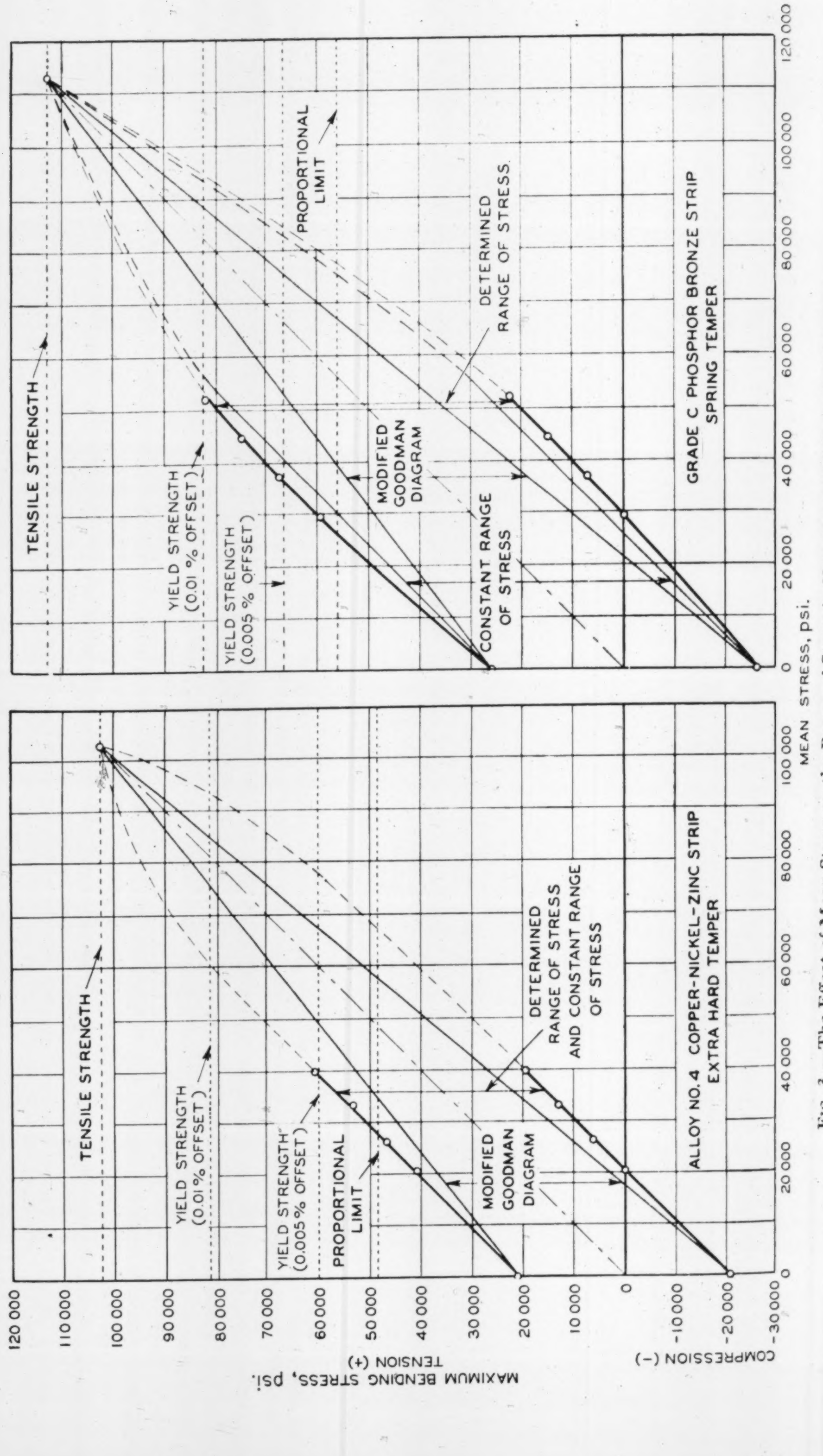

FIG. 3.—The Effect of Mean Stress on the Range of Stress in Unsymmetrical Repeated Bending.

This arm is connected to the frame on which the clamping blocks are mounted by two pivot arms *F*. The deflecting cam *G* together with the link *H* connecting the cam to the deflecting arm are adjustable to obtain different maximum deflections. A counter mechanism is attached to the motor shaft to record the number of cycles endured. The capacity of the machine is 20 test specimens, five of which were deflected from a position of no initial deflection to a maximum value, five from an initial deflection of 0.100 in. to a maximum, five from a deflection of 0.200 in. to a maximum, and five from a deflection of 0.300 in. to a maximum. All 20 of the specimens were deflected from these initial settings through an increment of strain corresponding to twice the eccentricity of the deflecting cam. Two such machines were used to determine the relationship between range of stress and mean stress for maximum stresses within the yield strength of the materials.

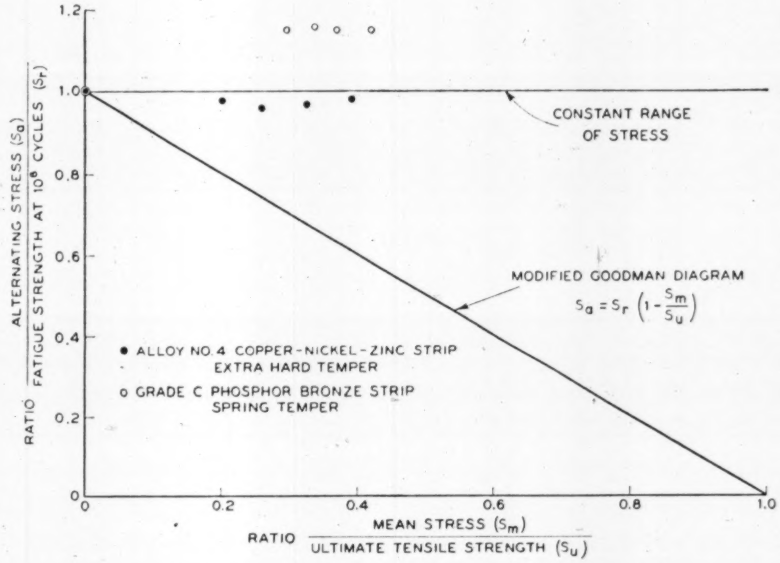

FIG. 4.—Alternating Stress-Mean Stress Diagram for Ranges of Repeated Bending Stresses.

The maximum fiber stresses in the test specimens corresponding to various deflections of the free end were computed on the basis of the load required to produce a given deflection and the moment arm corresponding to that load, that is from the basic stress formula

$$S_{\max} = \frac{M_y}{I} \ldots\ldots\ldots\ldots (2)$$

where

$S_{\max}$ = the maximum (numerical) bending stress of a range of stress in pounds per square inch,

M = the bending moment in inch-pounds,

y = the distance in inches between the neutral axis and the extreme fibers, and

I = the moment of inertia of the area of the section about the neutral axis (inches^4).

DISCUSSION OF RESULTS

The data obtained from the fatigue tests are given in Table III. The spread of these data is illustrated in Fig. 2 in which are plotted the *S-N* curves corresponding to the minimum and average values of fatigue strength. Numerical values are also indicated on the curves to show the constant minimum stresses used in the various unsymmetrical loading tests. The *S-N* curves for complete reversal of stress are likewise shown in Fig. 2.

To facilitate the discussion of the fatigue strength of the two materials at 10^8 cycles of stress under various loading conditions, modified Goodman diagrams have been constructed in Fig. 3. On these diagrams, lines corresponding to a constant range of stress are also shown. (In the case of the diagram for the copper-nickel-zinc alloy the constant range of stress lines are coincident with the lines drawn through the experimental data.) The circles represent the experimentally determined values, and it is immediately evident that the data correspond more nearly to a constant range of alternating stress than to the modified Goodman analysis.

The data for both materials are again represented in Fig. 4, which is an alternating stress - mean stress diagram in which the ratio S_a/S_r is plotted as a function of the ratio S_m/S_u. The inclined straight line represents the modified Goodman equation while the horizontal line represents a constant range of stress. Within the yield strength, for the two materials investigated, the experimental data more nearly correspond to the horizontal line representing a constant range of stress than to the sloping line representing the modified Goodman equation.

In comparison with Smith's data, (**9**) the findings for the two materials studied indicate that unsymmetrical stressing in bending is similar to that in torsion or in axial tension-compression when the mean stress is compression.

Experience in the design and use of non-ferrous spring materials has also confirmed this conclusion. Six years ago, derived constant range of stress diagrams were developed for various non-ferrous strip materials, based upon the fatigue data reported by Greenall and Gohn (**2**) for conditions of complete reversal of stress. These diagrams for Alloy No. 4 copper-nickel-zinc,[3] Grades A and C phosphor bronze,[4] type A copper-silicon alloy[5], heat-treated beryllium-copper,[6] alloy No. 8 brass,[7] and other materials in strip form in various tempers were used in the design of small flat springs which successfully withstood the required service conditions as determined both by accelerated laboratory and actual field tests.

CONCLUSION

For the two materials whose fatigue characteristics were determined for conditions of unsymmetrical bending, the limiting alternating stress component was constant for various mean stresses provided the maximum stress did not exceed the yield stress. In other words, the range of alternating stress (that is, the difference between the limiting maximum and minimum stresses) was constant irrespective of the mean stress so long as the maximum stress was less than the yield strength of the material. A further study of other materials in strip form is desirable to determine whether this relationship is general.

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[5] Standard Specifications for Copper Silicon Alloy Sheet and Strip for General Purposes (B 97 - 46), 1946 Book of A.S.T.M. Standards, Part I-B, p. 84.

[6] Tentative Specifications for Beryllium-Copper Alloy Strip (B 194 - 46a T), 1946 Book of A.S.T.M. Standards, Part I B, p. 414.

[7] Tentative Specifications for Brass Sheet and Strip (B 36 - 46 T), 1946 Book of A.S.T.M. Standards, Part I-B, p. 370.

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(1) J. R. Townsend and C. H. Greenall, "Fatigue Studies of Non-Ferrous Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 353 (1929).

(2) C. H. Greenall and G. R. Gohn, "Fatigue Properties of Non-Ferrous Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 160 (1937).

(3) G. R. Gohn and S. M. Arnold, "The Fatigue Properties of Beryllium-Copper Strip and Their Relation to Other Physical Properties," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 741 (1946).

(4) W. B. Price and R. W. Bailey, "Fatigue Properties of Five Cold Rolled Copper Alloys," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Inst. Metal Div., Vol. 124, p. 271 (1937).

(5) John Goodman, "Mechanics Applied to Engineering," Longmans, Green and Co., London, 9th Edition, p. 634 (1914).

(6) J. B. Johnson, "Materials of Construction," Fifth Ed., John Wiley and Sons, New York, N. Y., p. 781.

(7) H. F. Moore and J. B. Kommers, "Fatigue of Metals," McGraw-Hill Book Co., New York, N. Y., p. 173 (1927).

(8) J. B. Kommers, "The Effect of Range of Stress and Kind of Stress on Fatigue Strength," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 272 (1930).

(9) James O. Smith, "The Effect of Range of Stress on the Fatigue Strength of Metals," Engineering Experiment Station, University of Illinois, *Bulletin No. 334*, Vol. 39, No. 26 (1942).

(10) E. Orowan, "Theory of Fatigue Failure," *Proceedings*, Royal Society, Vol. 171, p. 79 (1939).

DISCUSSION

Mr. John N. Kenyon.[1]—Is it possible to obtain any information on the fatigue properties of the metals above the elastic limit? Plastic flow is a function of time and conceivably the cyclical stresses could be of such a high order of frequency that the metal would behave as though it were perfectly elastic. This question is prompted by the fact that the yield point can be exceeded under certain unpredictable variable stress conditions.

Mr. T. J. Dolan.[2]—The subject covered by Messrs. Gohn and Ellis is a rather active one about which we need further information. There appears to be a great deal of very confusing information in the literature when you try to survey the results of fatigue tests employing different types of stressing action or different kinds of material. Probably the effective range of stress that can be developed without fracture is influenced not only by the type of stressing action but also by the type of material itself, which is involved in the test. In Smith's paper, which has been referred to, it is proposed that the materials be divided into ductile and brittle classifications, and that also the effect of the state of stress be taken into consideration as affecting range of stress. It appears that for torsional stresses where the maximum shear stress and maximum tensile stress are equal and where no localized stresses are present that a constant range of stress seems to apply over a wide range of mean stresses. However, for the same material subjected to the same stressing condition, if the specimen is notched, then the modified Goodman diagram appears to apply.

In tests run just before the war on two aluminum alloys,[3] it was found that for notched specimens of the aluminum alloys in repeated bending tests, the modified Goodman diagram appeared to apply for one alloy but was on the unsafe side for the other alloy. That is in contrast with the data presented by the authors on bending, but, again, the fatigue behavior may be influenced considerably by the state of stress at the root of a very severe notch.

I should like to propose one possible explanation for some of these differences. In tests of the type made by the authors, employing unnotched specimens of thin sheet metal, the specimens are rather wide compared with their depth. The resulting stressing action is something more nearly approximating that of a slab rather than of a beam. The lateral stresses at right angles to the plane of the bending moment may, in themselves, set up a state of stress which is approximating that of torsion. The presence of a lateral compression and a longitudinal tension would make the authors' data fit in well with torsional test results having constant range of stress for various mean stresses.

Mr. R. E. Peterson.[4]—The authors

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[1] Research Engineer, Columbia University, New York, N. Y.

[2] Research Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[3] Thomas J. Dolan, "Certain Mechanical Strength Properties of Aluminum Alloys 25S-T and X76S-T," Nat. Advisory Comm. Aeronautics *Technical Note No. 914* (1943).

[4] Manager, Mechanics Div., Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

find a substantially constant range of stress as the mean stress is varied. It is of interest to compare this result with conclusions drawn from failure theory. As mentioned by the authors, Orowan's theory leads to a constant range of stress. It is interesting to note that Freudenthal[5] arrives at the same result, although his theory, which is based on probability, is quite different from Orowan's theory, which is based on cold working.

As the mean stress exceeds the yield strength and approaches the tensile strength, one would not expect the range to remain constant. A survey of data by J. O. Smith[6] shows that in torsion a constant range is maintained up to approximately the yield strength, but in bending the range seems to fall off as a function of steady stress along a sort of elliptic curve.

MR. H. J. GROVER.[7]—During the war, we made axial loading fatigue tests of sheet specimens of several high-strength aluminum alloys. These tests covered a considerable range of mean stress although they did not include completely reversed stress.

In general, the results of these tests varied between the two extremes mentioned by the authors of the present paper. At long lifetime (say upward of one million cycles), the range of stress appeared rather insensitive to variation of the mean stress up to nearly the tensile strength of the material. For short lifetimes (say ten thousand cycles), the range of stress diminished markedly with increasing mean stress.

Rather similar results appeared to exist for some magnesium sheet alloys for which we had much less extensive test data.

MR. W. C. ELLIS (*author's closure*).—At the outset I wish to express the appreciation of the authors for the interesting points that have been brought out in the discussion of this paper. It indicates the importance of studies of this type in the general field of fatigue testing.

In answer to Mr. Kenyon's question, with the machine which we used it was not possible to extend the tests far into the plastic range because of geometrical considerations. In addition such an extension introduces a serious problem in establishing a value for stress since the elastic theory is not valid. The test specimen also undergoes gross plastic extension in the critical section, at least during the initial cycles of stress, and the resulting strain adjustments introduce further uncertainties. In the usual design problem, the yield strength is controlling in those cases where the fatigue strength exceeds the yield strength.

With respect to fatigue tests above the elastic limit, we make a great many on lead alloys. Here the speed of testing is important for the very reason that plastic deformation or creep is occurring during the stress cycle. The results obtained depend greatly upon the rate of the stress cycle; the life is very much greater for high rates of stressing.

Mr. Dolan's remarks serve to emphasize the complexities which are encountered in tests run under conditions of unsymmetrical stressing with ductile and brittle materials and with notching. The explanation which he offers for the authors' results for strip in bending being the same as others have obtained in torsion seems reasonable. The authors hope that Mr. Dolan and others will develop such theoretical considerations further. This may go far toward resolving the differences in results obtained with different types of loading and conditions of specimens.

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[5] A. Freudenthal, "The Statistical Aspect of Fatigue of Metals," *Proceedings*, Royal Soc. (London), Series A, Vol. 187, p. 416 (1946).

[6] J. O. Smith, "Effect of Range of Stress on Fatigue Strength of Metals," University of Illinois Experiment Station, *Bulletin No. 334*, Vol. 39, No. 26 (1942).

[7] Battelle Memorial Institute, Columbus, Ohio.

Mr. Peterson's discussion is particularly appropriate as it serves to emphasize the theoretical analyses which have been made of the effects of cyclic stressing. It is encouraging that Orowan and Freudenthal predict from different models so many of the characteristic property dependencies observed in experimental fatigue results.

The findings for axial loading tests on high-strength aluminum alloys reported by Mr. Grover add to the discussion in that he considered the effects of unsymmetrical stressing on stress range for short life times and found that under these conditions a constant range of stress did not prevail. The authors have not analyzed their results for short lifetimes, although the data of Fig. 4 admit of such an analysis for lifetimes to a minimum of 500,000 hr. The summary which Mr. Grover has given of his work points out again the need for further effort in the field of unsymmetrical stressing before the situation is clearly rationalized.

THE CREEP CHARACTERISTICS OF COPPER AND SOME COPPER ALLOYS AT 300, 400, AND 500 F.*

By H. L. Burghoff[1] and A. I. Blank[1]

Synopsis

This paper presents additional information obtained in a continuing program of creep testing of wrought copper alloys at 300, 400, and 500 F. Creep data including total creep, creep rates, and relative creep strengths, are shown for four types of copper and several copper alloys in annealed and hard-drawn tempers. Tensile properties and notations on microstructure of the test materials before and after creep testing are given.

The four types of copper which were tested conform to the following order of decreasing creep strength in both annealed and hard-drawn tempers: arsenical copper, deoxidized copper, oxygen-free copper, and electrolytic copper. The superiority of the arsenical copper over the other coppers is pronounced. The difference between oxygen-free and electrolytic coppers is very slight.

The creep strengths of admiralty and aluminum brass in the annealed condition vary with grain size. While there are some significant differences in the creep behavior of these two alloys in hard-drawn tempers, it appears that the presence of the tin and of the aluminum in these special brasses contributes little to creep strength for the annealed condition.

Two age-hardenable alloys, containing copper, nickel and phosphorus and copper, nickel, phosphorus and tellurium, respectively, display high resistance to creep. As age-hardened and not subsequently cold-drawn, they are rendered brittle for conditions of exposure which are severe with respect to stress and temperature. The copper-nickel-phosphorus alloy as severely cold drawn after age-hardening is much more stable.

Additional data are included to amplify previously published information on red brass, naval brass, phosphor bronze, and 3 per cent silicon bronze. The creep behavior of the copper-base materials thus far tested appears to be intimately associated with stability as indicated by softening and recrystallization characteristics of hard-drawn tempers at the testing temperatures.

Two papers (**1, 2**)[2] have already been presented by the authors on the creep characteristics of one type of copper and some copper alloys at 300, 400, and 500 F. The present paper deals with the creep characteristics of three other types of copper and several other copper alloys, and also adds further information on the previously reported materials. These three papers represent the results of a continuing program to determine the creep characteristics of wrought copper-base materials as affected by composition, temper, and temperature.

Since publication of the two earlier papers (**1, 2**), several other papers

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Research Metallurgist and Assistant Research Metallurgist, respectively, Chase Brass and Copper Co., Waterbury, Conn.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 753.

TABLE I.—COMPOSITIONS AND PROPERTIES OF MATERIALS TESTED.

Material	Analysis by Weight, per cent	Temper	Yield Strength, 0.5 per cent Elongation under Load, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent
Electrolytic copper	99.96 Cu; <0.002 Fe; <0.002 Ni; <0.001 Pb; <0.0005 Sn	Annealed, 0.025-mm. grain size	7 200	35 100	50.0
		Drawn 84 per cent	50 000	55 400	11.0
Oxygen-free copper	99.98 Cu; <0.01 Zn; <0.003 Fe; <0.005 Ni; <0.0005 Pb	Annealed, 0.025-mm. grain size	7 900	34 500	50.0
		Drawn 84 per cent	49 500	54 500	9.0
Deoxidized copper	99.95 Cu; 0.008 P; 0.001 Fe; 0.001 Ni	Annealed, 0.013-mm. grain size	12 000	36 500	48.0
		Annealed, 0.013-mm; and Stretched 1 per cent	15 400	37 000	43.0
		Annealed, 0.013-mm; and Stretched 6 per cent	33 000	41 300	29.5
Arsenical copper	99.60 Cu; 0.32 As; 0.022 P; 0.015 Fe; 0.005 Ni; 0.0005 Pb; <0.01 Zn; <0.002 Sb; <0.001 Mn	Annealed, 0.045-mm. grain size	6 100	35 000	49.0
		Drawn 84 per cent	55 500	60 900	11.0
Red brass	84.79 Cu; 0.004 Fe; 0.002 Pb; 15.20 Zn[a]	Annealed, 0.060-mm. grain size	9 000	41 000	47.0
		Drawn 37 per cent	59 000	67 500	10.5
		Drawn 84 per cent	67 000	96 500	7.0
Admiralty	71.06 Cu; 0.92 Sn; 0.008 Fe; 0.015 Pb; 0.003 Ni; 27.99 Zn[a]	Annealed, 0.055-mm. grain size	15 500	52 000	60.0
		Annealed, 0.018-mm. grain size	26 000	57 000	52.0
		Drawn 60 per cent	71 000	109 000	6.0
Aluminum brass	76.43 Cu; 2.01 Al; 0.01 Fe; <0.005 Pb; 21.55 Zn[a]	Annealed, 0.030-mm. grain size	23 500	58 100	53.0
		Annealed, 0.015-mm. grain size	27 500	60 600	47.0
		Drawn 37 per cent	61 500	86 800	12.0
Naval brass	59.98 Cu; 0.65 Sn; 0.015 Fe; 0.0005 Bi; 39.35 Zn[a]	Annealed at 1000 F.	31 500	64 500	40.0
		Drawn 37 per cent	66 000	95 000	10.0
3 per cent silicon bronze	96.34 Cu; 2.80 Si; 0.06 Fe; 0.005 Pb; 0.80 Zn[a]	Annealed, 0.100-mm. grain size	8 000	49 500	65.0
		Annealed, 0.008-mm. grain size	25 000	61 500	61.0
		Drawn 37 per cent	58 000	90 500	9.0
Phosphor bronze	94.16 Cu; 5.56 Sn; 0.24 P; 0.01 Fe; 0.005 Ni; 0.002 Bi	Annealed, 0.050-mm. grain size	20 000	52 000	66.0
		Drawn 84 per cent	77 500	144 000	3.5
Cu-Ni-P alloy	98.57 Cu; 1.15 Ni; 0.23 P; 0.005 Fe; <0.01 Pb, Sn, Zn.	Quenched from 1450 F. and Aged 4 hr. at 850 F.	58 500	72 000	18.0
		Quenched from 1450 F., Aged 4 hr. at 850 F., and Drawn 84 per cent	80 000	100 000	5.5
Cu-Ni-P-Te alloy	98.09 Cu[a]; 1.11 Ni; 0.51 Te; 0.28 P; 0.003 Fe; 0.005 Pb; <0.01 Zn.	Quenched from 1450 F. and Aged 1.5 hr. at 850 F.	53 000	64 900	20.0
		Quenched from 1450 F., Drawn 30 per cent, and Aged 1.5 hr. at 770 F.	70 000	80 500	12.5

[a] By difference.

dealing with creep characteristics of copper and copper alloys have appeared in the literature. Included among these are publications by E. A. Davis (3), C. H. M. Jenkins, E. H. Bucknall and E. A. Jenkinson (4), and D. J. McAdam, Jr., G. W. Geil and D. H. Woodard (5).

TEST MATERIALS

The previous work by the authors on admiralty has been expanded to include another annealed temper, and there are also additional test results listed for red brass, naval brass, phosphor bronze,

TABLE II.—EFFECT OF CREEP TEST EXPOSURES ON ROOM TEMPERATURE PROPERTIES.

Temper	Creep Testing			Tensile Strength, psi.	Elongation in 2 in., per cent	Microstructure
	Temperature, deg. Fahr.	Stress, psi.	Time, hr.			
ELECTROLYTIC COPPER						
Drawn 84 per cent	(As processed)			55 400	11	Hard drawn
	300	0	24	55 000	11	Unchanged
	300	0	6500	45 000	22	About 50 per cent recrystallized
	300	10 000	6500	44 600	21	About 50 per cent recrystallized
	300	20 000	6500	43 100	25	About 50 per cent recrystallized
	400	0	24	43 500	25	Partially recrystallized
	400	0	240	37 400	37	Completely recrystallized
	400	0	6500	35 900	45	Completely recrystallized
	400	1 050	6500	36 100	46	Completely recrystallized
	400	7 100	6500	36 000	43	Completely recrystallized
	500	0	24	36 200	47	Completely recrystallized
OXYGEN-FREE COPPER						
Drawn 84 per cent	(As processed)			54 500	9	Hard-drawn
	300	0	24	55 300	11	Unchanged
	300	0	6500	45 200	24	About 25 per cent recrystallized
	300	9 950	6500	44 400	24	About 25 per cent recrystallized
	300	20 000	5700	42 600	25	About 25 per cent recrystallized
	400	0	24	45 000	24	Partially recrystallized
	400	0	240	36 800	43	Completely recrystallized
	400	0	6500	35 500	45	Completely recrystallized
	400	1 050	6500	35 400	44	Completely recrystallized
	400	7 000	6500	35 700	45	Completely recrystallized
DEOXIDIZED COPPER						
Annealed, 0.013 mm. and Stretched 6 per cent	(As processed)			41 300	29	Alpha grains
	300	12 100	6400	42 000	28	Unchanged
	400	2 050	6000	38 300	35	Apparently unchanged
	400	4 000	6000	37 700	33	Apparently unchanged
	400	8 000	6000	37 300	38	Apparently unchanged
	500	1 050	6000	35 600	39	Some recrystallization to about 0.100-mm. grain size
	500	3 100	6000	35 000	38	
ARSENICAL COPPER						
Drawn 84 per cent	(As processed)			60 900	11	Hard drawn
	300	0	24	61 200	12	Unchanged
	300	0	6500	57 600	11	Unchanged
	300	12 400	6400	59 500	10	Unchanged
	300	30 100	6500	58 900	11	Unchanged
	400	0	24	59 600	12	Unchanged
	400	0	6500	56 700	14	Unchanged
	400	5 050	6500	56 800	12	Unchanged
	400	20 100	6500	54 200	14	Unchanged
	500	0	24	58 100	15	Unchanged
	500	0	6500	40 700	36	Completely recrystallized
	500	2 050	6000	41 700	37	Completely recrystallized
	500	5 000	6500	41 300	38	Completely recrystallized
RED BRASS						
Drawn 84 per cent	(As processed)			96 500	7	Hard drawn
	300	0	24	96 500	7	Unchanged
	300	0	5100	94 600	7	Unchanged
	300	19 800	5100	94 300	6	Unchanged
	300	53 800	5100	92 400	6	Unchanged

Continued on pages 728 and 729.

TABLE II—*Continued.*

Temper	Creep Testing			Tensile Strength, psi.	Elongation in 2 in., per cent	Microstructure
	Temperature, deg. Fahr.	Stress, psi.	Time, hr.			
ADMIRALTY						
Drawn 60 per cent	(As processed)			109 000	6	Hard drawn
	300	0	24	110 300	4	Unchanged
	300	0	6500	108 300	4	Unchanged
	300	15 000	6500	109 900	3	Unchanged
	300	40 650	5200	107 500	4	Unchanged
	400	0	24	106 900	3	Unchanged
	400	0	5750	78 000	19	About 75 per cent recrystallized
	400	2 000	5750	80 100	15	About 75 per cent recrystallized
	400	9 850	5750	77 500	19	About 75 per cent recrystallized
	500	0	24	67 900	31	More than 90 per cent recrystallized
	500	0	240	64 600	43	Completely recrystallized
	500	0	5750	63 000	43	Completely recrystallized
	500	300	5750	62 700	41	Completely recrystallized
	500	2 000	5300	62 700	41	Completely recrystallized
ALUMINUM BRASS						
Drawn 37 per cent	(As processed)			86 800	12	Hard drawn
	300	0	24	88 600	10	Unchanged
	300	0	6500	89 000	8	Unchanged
	300	30 100	6500	87 000	9	Unchanged
	300	50 000	6500	89 500	6	Unchanged
	400	0	24	87 200	9	Unchanged
	400	0	6500	87 900	10	Unchanged
	400	15 000	6000	86 100	12	Unchanged
	400	30 300	6000	86 400	9	Unchanged
	500	0	24	87 300	12	Unchanged
	500	0	240	74 400	24	Partially recrystallized
	500	0	6000	63 200	43	Completely recrystallized
	500	2 050	6000	63 000	45	Completely recrystallized
	500	3 050	6000	62 700	44	Completely recrystallized
COPPER-NICKEL-PHOSPHORUS ALLOY						
Quenched from 1450 F. and Aged 4 hr. at 850 F.	(As processed)			72 000	18	Alpha grains
	300	0	5900	71 000	17	Unchanged
	300	20 200	5150	71 300	18	Unchanged
	300	40 200	5900	70 000	16	Unchanged[a]
	300	45 600	4030	68 000	12	Some intergranular separation
	400	0	5400	71 100	17	Unchanged
	400	7 200	5400	71 600	15	Unchanged
	400	10 050	5050	71 000	15	Unchanged
	400	15 250	1600	70 300	15	Unchanged
	500	0	5750	71 200	19	Unchanged
	500	3 050	5100	71 800	17	Unchanged
	500	5 100	5100	68 100	9	Unchanged[a]
	500	5 900	5400	70 300	15	Unchanged[a]
	500	8 100	5250	56 300	7	Unchanged[a]
	500	10 150	5750	49 000	2	Unchanged[a]
Quenched from 1450 F., Aged 4 hr. at 850 F., and Drawn 84 per cent	(As processed)			100 000	5	Hard drawn
	300	0	24	100 300	5	Unchanged
	300	0	5900	97 300	7	Unchanged
	300	25 000	5150	96 000	5	Unchanged
	300	69 900	5900	96 700	7	Unchanged
	400	0	24	98 500	7	Unchanged
	400	0	5400	95 400	7	Unchanged
	400	21 100	5400	93 400	6	Unchanged
	400	39 800	5100	94 300	6	Unchanged
	500	0	24	96 600	10	Unchanged
	500	0	5400	89 500	10	Unchanged except for fewer strain lines
	500	10 250	5100	89 200	8	Unchanged except for fewer strain lines
	500	35 000	5400	88 200	8	Unchanged except for fewer strain lines
	500	49 200	4400	86 900	8	Unchanged except for fewer strain lines

TABLE II.—*Concluded.*

Temper	Creep Testing			Tensile Strength, psi.	Elongation in 2 in., per cent	Microstructure
	Temperature, deg. Fahr.	Stress, psi.	Time, hr.			
			Copper-Nickel-Phosphorus-Tellurium Alloy			
Quenched from 1450 F. and Aged 1.5 hr. at 850 F.	(As processed)			64 900	20	Alpha grains plus telluride
	300	0	6000	65 900	20	Unchanged
	300	20 700	6000	65 200	20	Unchanged
	300	41 500	6000	65 000	19	Unchanged
	400	0	6000	65 400	19	Unchanged
	400	10 000	6000	64 600	21	Unchanged
	400	20 150	6000	65 000	20	Unchanged[a]
	500	0	6000	60 000	11	Unchanged[a]
	500	5 050	6000	58 200	8	Unchanged[a]
	500	10 150	6000	49 000	5	Unchanged[a]
Quenched from 1450 F., Drawn 30 per cent and Aged 1.5 hr. at 770 F.	(As processed)			80 500	12	Hard drawn
	300	0	6000	81 400	12	Unchanged
	300	19 900	6000	80 800	13	Unchanged
	300	39 800	6000	82 200	13	Unchanged
	300	48 800	6000	80 000	12	Unchanged
	300	59 800	5800	73 500	2	Some intergranular separation
	400	0	6000	82 000	13	Unchanged
	400	10 000	6000	81 100	14	Unchanged
	400	15 000	6000	81 400	13	Unchanged
	400	20 400	6000	78 700	3	Unchanged[a]
	400	30 100	6000	73 200	2	Unchanged[a]
	500	0	6860	80 500	12	Unchanged
	500	5 950	6150	80 500	11	Unchanged[a]
	500	10 200	6860	78 500	8	Unchanged[a]

[a] Some intergranular separation at and immediately adjacent to fracture in tension test after creep-test exposure.

and 3 per cent silicon bronze for conditions which were covered less extensively or not at all in the earlier paper. The work on deoxidized copper now includes creep data for the material as stretched 1 and 6 per cent after annealing. The materials which are included for the first time are electrolytic copper, oxygen-free copper (OFHC), arsenical copper, aluminum brass, and age-hardenable copper-nickel-phosphorus and copper-nickel-phosphorus-tellurium alloys. Composition, temper and tensile properties of all the test materials are listed in Table I. All of these materials were processed and tested in the form of straight lengths of $\frac{1}{8}$-in. diameter wire.

Procedure

The multiple specimen furnaces, in which the creep tests were performed, have been previously described (1). In the present work the only change in the established testing procedure concerns the method of measuring the creep of the specimens. The optical system which was previously used to measure creep by sighting with a cathetometer on reference blocks attached to each specimen has been replaced by an indicating system which consists of a dial indicator attached to each specimen. Each indicator is located outside the furnace and is secured by two cupronickel extension rods, one of which is attached to the body of the indicator and also to one reference point on the test specimen and the other of which is attached to the plunger of the indicator and also to a second reference point on the test specimen. The extension of the specimen within the reference length, which is 10 in., is thus transferred to and indicated by the dial indicator. The indicators, which are graduated in 0.0001-in. divisions, are much more easily read than the cathetometer, are fully as sensitive and accurate, and permit the accumulation of many more points on the creep curve than was hitherto feasible. The fact that the two extension rods are very close to each other and to the test specimen minimizes the possibility of temperature differences at corresponding points on the extension rods and specimen. Any small differences in thermal expansion and contraction of the specimen and the extension rods due to temperature fluctuations and slight differences in coefficients of thermal expansion have been found by calculation to be negligibly small.

DISCUSSION OF TEST RESULTS

Typical values of tensile strength and elongation for the materials as determined at room temperature after creep test exposures are listed in Table II together with notations on microstructure. Comparison of this information with that pertaining to the materials as processed indicates the type and extent of any changes which may have occurred during the creep tests. The tensile properties for most of the hard-drawn materials after exposure at the test temperatures for 24 hr. are also listed in Table II in order to provide a measure of any possible change that may have occurred prior to the application of load at the beginning of the creep test, since all specimens were held at temperature for 24 hr. before loading to establish temperature equilibrium.

Table III gives details concerning creep testing temperature, creep stress, duration of test, initial extension, total creep, creep intercept, e_0, and creep rate, v, in the latter part of the test, usually at about 5000 to 6000 hr. The values e_0 and v may be applied to the McVetty type of formula for calculating amount of creep (6):

$$\text{Creep} = e_0 + vt$$

where

e_0 = a constant (intercept for 0 time on creep-time plot),
v = creep rate (slope or tangent to creep curve), and
t = time.

The stresses required to produce some or all of the creep rates of 0.001, 0.01, and 0.1 per cent per 1000 hr., as derived from the creep test results, are listed in Table IV. They constitute a simple means of comparison of the relative creep strengths of the various materials.

The relationships between stress and creep rate for the test materials are plotted logarithmically in several graphs. The creep rates are those listed in Table III. For the most part they represent creep which was still progressing at a decreasing rate at the end of the tests. A steady rate of creep was attained in relatively few cases. In those instances where creep rates were accelerating, the plotted points are underlined. For a more concise comparison of creep strengths, the stresses required to produce a creep rate of 0.01 per cent per 1000 hr. have been plotted as a function of temperature.

The majority of the creep tests in this work did not progress beyond the first stage of creep, that of decreasing creep rate, within the usual test period of about 6000 hr. In many instances the creep rates were still decreasing even after considerably longer periods of time. Two of the tests listed in Table III are selected to illustrate the continuing decrease of creep rate over very long periods of time. The first is for a stress of 25,000 psi. at 300 F. on aluminum brass as annealed to 0.015-mm. grain size. Its creep rate in per cent per 1000

TABLE III.—RESULTS OF CREEP TESTS.

Temper	Testing Temperature, deg. Fahr.	Stress, psi.	Total Time, hr.	Initial Extension, per cent	Total Creep, per cent	Creep	
						Intercept, e_0, per cent	Rate, v, per cent per 1000 hr.
			ELECTROLYTIC COPPER				
Annealed 0.025-mm. grain size	300	2 050	6400	0.020	0.068	0.048	0.0032
		3 000	6500	0.037	0.220	0.133	0.013
		6 000	6500	0.385	1.490	1.120	0.0575
		8 100	6500	1.110	2.365	1.795	0.088
	400	1 550	6000	0.018	0.150	0.067	0.014
		2 050	6500	0.023	0.336	0.168	0.026
		3 050	6000	0.040	1.010	0.510	0.083
		4 000	6500	0.090	1.952	1.232	0.11
		6 100	6000	0.595	1.890	0.668	0.204
		7 050	4500	0.950	2.950	2.750	0.267
	500	360	6000	0.003	0.081	0.016	0.011
		600	6000	0.005	0.190	0.010	0.030
		1 050	6500	0.010	0.630	0.113	0.0795
		2 000	6500	0.020	2.857	0.869	0.306
rawn 84 per cent	300	7 550	6400	0.045	0.073	0.041	0.0049
		10 000	6500	0.060	0.107	0.042	0.010
		14 650	6400	0.090	0.450	−0.170	0.097[a]
		20 000	6500	0.130	2.20	−3.00	0.80[a]
		25 200	1780	0.185	2.38	−4.98	4.14[a]
	400	1 050	6500	0.012	0.052	0.045	0.0011
		2 100	6000	0.022	0.181	0.112	0.0115
		4 050	6500	0.041	1.039	0.409	0.097
		7 100	6500	0.078	5.34	2.47	0.44
			OXYGEN-FREE COPPER				
Annealed, 0.025-mm. grain size	300	2 100	6400	0.019	0.034	0.024	0.0017
		3 050	6500	0.030	0.098	0.049	0.0075
		4 550	6000	0.080	0.430	0.290	0.023
		6 000	6500	0.263	1.107	0.847	0.040
		7 950	5100	0.510	1.980	1.560	0.083
	400	1 300	6000	0.011	0.061	0.022	0.0065
		2 050	6500	0.020	0.193	0.054	0.021
		3 100	6000	0.030	0.550	0.256	0.049
		4 050	6500	0.060	1.235	0.725	0.078
		6 100	6500	0.370	1.089	0.309	0.12
		7 400	5000	0.830	3.750	2.670	0.215
	500	350	6000	0.006	0.036	−0.018	0.0089
		600	6000	0.010	0.1175	0.0135	0.017
		1 050	6500	0.013	0.374	0.052	0.0495
		2 000	6000	0.024	1.126	0.344	0.13
Drawn 84 per cent	300	7 900	6000	0.051	0.051	0.002	0.0082
		7 900	9430	0.051	0.079	0.002	0.0082
		9 950	6500	0.065	0.102	−0.041	0.022[a]
		20 000	5700	0.134	2.44	−5.33	1.36[a]
		25 100	3050	0.172	2.41	−9.42	3.88[a]
	400	1 050	6500	0.010	0.049	0.042	0.0011
		2 100	3200	0.022	0.135	0.090	0.014
		3 050	6000	0.030	0.392	0.185	0.0345
		4 050	6100	0.043	0.955	0.506	0.074
		5 600	6000	0.062	2.015	1.190	0.14
		7 000	6500	0.080	3.72	2.50	0.19
			DEOXIDIZED COPPER				
Annealed 0.013-mm. grain size and stretched 1 per cent	300	3 700	6400	0.022	0.042	0.025	0.0027
		5 150	6500	0.028	0.081	0.031	0.0077
		7 900	6400	0.050	0.285	0.106	0.028
		10 600	6500	0.074	1.180	0.763	0.064
	400	2 100	6500	0.017	0.101	0.058	0.0066
		3 600	6000	0.030	0.248	0.091	0.026
		5 150	6500	0.044	0.707	0.244	0.071
	500	1 100	6100	0.010	0.214	0.071	0.0235
		3 100	4550	0.027	1.440	0.352	0.24

[a] Third stage of creep, accelerating creep rate.

Continued on pages 732, 733, 734, and 735.

TABLE III.—*Continued.*

Temper	Testing Temperature, deg. Fahr.	Stress, psi.	Total Time, hr.	Initial Extension, per cent	Total Creep, per cent	Creep	
						Intercept, e_0, per cent	Rate, v, per cent per 1000 hr.
			DEOXIDIZED COPPER (*Continued*)				
Annealed 0.013-mm. grain size and stretched 6 per cent	300	8 900	6000	0.065	0.077	0.055	0.0037
		12 100	6400	0.090	0.115	0.076	0.0060
		17 250	5400	0.140	0.322	0.159	0.030
		20 100	6000	0.184	8.465	4.585	0.647
	400	2 050	6000	0.016	0.063	0.041	0.0037
		3 000	6000	0.025	0.145	0.071	0.0123
		4 000	6000	0.033	0.253	0.099	0.026
		8 000	6000	0.065	0.894	0.285	0.102
	500	1 050	6000	0.010	0.132	0.055	0.013
		2 050	6000	0.019	0.441	0.172	0.045
		3 100	6000	0.028	0.770	0.237	0.089
			ARSENICAL COPPER				
Annealed 0.045-mm. grain size	300	3 000	6500	0.042	0.013	0.013	<0.0001
		5 200	6400	0.480	0.100	0.085	0.00235
		7 200	6400	1.075	0.161	0.122	0.0062
		7 950	6500	1.320	0.240	0.185	0.0085
		10 250	6400	2.140	0.397	0.275	0.019
	400	2 050	6500	0.028	0.009	0.007	<0.001
		3 050	6000	0.065	0.054	0.040	0.0023
		4 000	6500	0.235	0.198	0.158	0.0062
		5 600	6000	0.675	0.380	0.295	0.014
		7 100	6000	1.150	0.498	0.365	0.022
	500	1 000	6500	0.010	0.017	0.014	0.0005
		2 050	6860	0.020	0.087	0.049	0.0055
		3 550	6860	0.180	0.498	0.335	0.024
		6 250	6000	0.975	1.609	0.700	0.152
Drawn 84 per cent	300	12 400	6400	0.070	0.019	0.017	0.00024
		20 150	6500	0.117	0.036	0.033	0.0005
		30 100	6500	0.183	0.099	0.089	0.0016
		40 000	6000	0.260	0.166	0.114	0.00875
		40 000	9450	0.260	0.192	0.118	0.0078
		47 200	500	0.380	0.370	0.160	0.420^b
		47 200	750	0.380	0.530	...	...
	400	5 050	6500	0.036	0.008	0.008	0.0001
		8 950	6000	0.062	0.035	0.032	0.0005
		15 250	6000	0.105	0.070	0.058	0.0019
		20 100	6500	0.138	0.088	0.070	0.0027
		30 500	6000	0.212	0.305	0.208	0.016
		40 000	360	0.298	0.67	0.252	1.18^b
		40 000	1850	0.298	4.76	...	...
	500	1 550	6000	0.018	0.011	0.0015	0.00145
		2 050	6000	0.016	0.059	−0.012	0.012^a
		3 550	7300	0.027	0.578	−0.187	0.105^a
		5 000	6500	0.037	0.896	−0.672	0.24^a
			RED BRASS				
Annealed 0.060 mm. grain size	300	6 960	5100	0.048	0.039	0.024	0.0029
	400	1 000	4400	0.010	0.012	0.008	0.0008
		3 500	5100	0.035	0.057	0.040	0.0033
		5 850	5100	0.058	0.120	0.026	0.019
		6 940	6600	0.070	0.412	0.187	0.034
		8 000	5250	0.235	1.031	0.670	0.069
		8 940	2000	0.460	0.600	0.390	0.10
		11 500	4130^c	1.700	1.920^c	0.750	0.283
	500	850	5140	0.011	0.031	0.010	0.0040
		980	6600	0.013	0.060	0.029	0.0046
		1 000	5140	0.013	0.038	0.011	0.0053
		2 000	5000	0.025	0.066	0.026	0.0080
		2 950	5140	0.035	0.109	0.024	0.0165
		3 520	5300	0.045	0.196	0.037	0.030
		4 980	5000	0.060	0.447	0.083	0.073
		5 780	5140	0.069	0.810	0.163	0.126
		6 000	5140	0.071	0.887	0.180	0.138
		6 950	1600	0.080	1.020	0.510	0.32

[b] This value applies to second stage of creep at indicated time Test entered third stage of creep thereafter and specimen failed with a ductile fracture shortly after maximum time indicated for test.

[c] Followed shortly by brittle failure.

TABLE III.—*Continued.*

Temper	Testing Temperature, deg. Fahr.	Stress, psi.	Total Time, hr.	Initial Extension, per cent	Total Creep, per cent	Creep	
						Intercept, e_0, per cent	Rate, v, per cent per 1000 hr.
			RED BRASS (*Continued*)				
Drawn 37 per cent	300	2 000	3700	0.012	0.002	0.002	<0.0001
		5 040	5200	0.030	0.020	0.018	0.0004
		13 050	5050	0.080	0.058	0.044	0.0028
		17 650	5100	0.110	0.117	0.083	0.0067
		23 850	6050	0.147	0.219	0.153	0.011
	400	4 980	5200	0.025	0.052	0.039	0.0026
		11 850	5200	0.067	0.216	0.146	0.013
Drawn 84 per cent	300	19 800	5100	0.130	0.067	0.054	0.0026
		53 800	5100	0.440	0.420	0.250	0.033
			ADMIRALTY				
Annealed 0.055-mm. grain size	400	1 970	5400	0.010	0.016	0.013	0.0005
		4 000	5400	0.020	0.026	0.019	0.0014
		5 820	6400	0.032	0.045	0.028	0.0027
		6 920	5200	0.040	0.042	0.022	0.0038
		9 960	5700	0.062	0.066	0.027	0.0067
		13 000	6600	0.091	0.164	0.044	0.018
Annealed 0.018-mm. grain size	300	1 980	3700	0.013	0.005	0.004	0.0002
		5 000	5100	0.034	0.023	0.018	0.0009
		9 840	5100	0.067	0.042	0.029	0.0025
		11 800	5200	0.080	0.061	0.044	0.0033
		14 950	5100	0.101	0.080	0.053	0.0053
		19 750	5100	0.134	0.125	0.073	0.010
		25 000	5900	1.05	0.525	0.230	0.050
	400	800	5100	0.005	0.022	0.009	0.0026
		1 970	5100	0.014	0.043	0.014	0.0056
		4 000	5100	0.030	0.136	0.047	0.0174
		6 940	5100	0.050	0.355	0.100	0.050
		10 500	5100	0.075	0.695	0.055	0.125
	500	480	5100	0.004	0.111	0.008	0.020
		1 020	5000	0.010	0.398	0.080	0.064
		2 000	5000	0.018	1.30	0.155	0.228
		4 000	5000	0.035	2.24	0.045	0.440
		5 860	2350	0.052	2.67	−0.66	1.43^a
Drawn 60 per cent	300	12 550	5200	0.090	0.074	0.066	0.0016
		15 000	6500	0.107	0.096	0.078	0.0027
		17 250	5200	0.125	0.103	0.089	0.0028
		20 100	5400	0.143	0.144	0.116	0.0052
		30 300	6500	0.215	0.236	0.179	0.0088
		40 650	5200	0.285	0.400	0.307	0.018
		50 200	5200	0.355	0.698	0.385	0.060
	400	2 000	5750	0.016	0.050	−0.016	0.0115^a
		4 000	5750	0.032	0.093	−0.017	0.019^a
		8 100	5750	0.065	0.241	−0.143	0.067^a
		9 850	5750	0.078	0.335	−0.278	0.106^a
	500	300	5750	0.004	0.084	0.052	0.0055
		490	6100	0.005	0.180	0.062	0.0192
		2 000	5300	0.021	2.460	0.160	0.435
			ALUMINUM BRASS				
Annealed 0.030-mm. grain size	300	5 900	5 500	0.030	0.0075	0.002	0.0010
		9 900	6 500	0.050	0.049	0.024	0.0038
		19 900	6 500	0.115	0.335	0.146	0.029
		22 000	6 000	0.130	0.668	0.321	0.058
		22 000	9 450	0.130	0.849	0.367	0.051
	400	2 000	6 500	0.018	0.056	0.028	0.0043
		5 000	6 500	0.041	0.145	−0.007	0.0235
		10 000	6 500	0.085	1.200	0.035	0.18
	500	550	6 860	0.005	0.092	0.043	0.0071
		1 050	6 860	0.010	0.288	0.082	0.030
		1 550	6 500	0.014	0.510	0.128	0.059

TABLE III.—*Continued.*

Temper	Testing Temperature, deg. Fahr.	Stress, psi.	Total Time, hr.	Initial Extension, per cent	Total Creep, per cent	Creep	
						Intercept, e_0, per cent	Rate, v, per cent per 1000 hr.
ALUMINUM BRASS (*Continued*)							
Annealed 0.015-mm. grain size	300	5 000	6 500	0.027	0.004	0.001	0.0005
		10 000	6 500	0.079	0.054	0.034	0.0031
		19 850	6 500	0.150	0.359	0.183	0.027
		25 000	6 000	0.205	1.045	0.515	0.088
		25 000	11 100	0.205	1.455	0.655	0.072
	400	2 050	6 500	0.013	0.060	0.030	0.00465
		5 050	6 500	0.030	0.312	0.094	0.0335
		10 100	6 500	0.060	1.580	0.143	0.22
	500	1 550	3 170	0.015	0.353	0.111	0.076
Drawn 37 per cent	300	19 150	6 400	0.116	0.092	0.074	0.0028
		30 100	6 500	0.180	0.169	0.128	0.0063
		39 900	6 000	0.245	0.285	0.190	0.016
		39 900	11 100	0.245	0.345	0.233	0.010
		50 000	6 500	0.322	0.847	0.498	0.054
	400	15 000	6 000	0.110	0.138	0.118	0.0033
		20 100	6 500	0.147	0.256	0.206	0.0077
		30 300	6 000	0.220	1.060	0.480	0.097
		35 200	4 130	0.255	2.585	0.485	0.51
	500	1 050	6 000	0.007	0.152	0.081	0.012
		2 050	6 000	0.014	0.563	0.197	0.061
		3 050	6 000	0.022	1.221	0.117	0.184
		5 050	3 480	0.032	2.290	−0.181	0.71
NAVAL BRASS							
Annealed at 1000 F.	300	3 100	5600	0.019	0.009	0.007	0.0004
		7 100	5600	0.046	0.031	0.024	0.0012
		20 000	6050	0.120	0.336	−0.072	0.0674[a]
Drawn 37 per cent	300	1 000	5600	0.008	0.016	0.012	0.0007
		2 050	5550	0.016	0.044	0.038	0.0011
		8 000	6000	0.061	0.151	0.124	0.0045
		24 900	5830	0.193	1.080	0.504	0.098
3 PER CENT SILICON BRONZE							
Annealed 0.100-mm. grain size	300	7 650	5000	0.055	0.007	0.006	0.0002
		10 100	4680	0.53	0.040	0.030	0.0011
		12 350	5400	1.10	0.047	0.023	0.0044
		13 900	5400	1.73	0.070	0.035	0.0064
		17 150	6000	3.45	0.225	0.105	0.020
Annealed 0.008-mm. grain size	300	14 950	5400	0.071	0.062	0.047	0.0027
		24 650	5400	1.52	1.08	1.02	0.011
Drawn 37 per cent	300	15 200	5900	0.110	0.054	0.053	<0.001
		24 750	5400	0.183	0.095	0.083	0.0023
		39 800	5400	0.296	0.205	0.156	0.0090
PHOSPHOR BRONZE							
Annealed 0.050-mm. grain size	400	5 870	5100	0.033	0.032	0.020	0.0024
		10 170	5100	0.057	0.068	0.041	0.0053
	500	720	5100	0.007	0.007	0.005	0.0004
		1 550	5100	0.014	0.025	0.017	0.0016
		2 470	5000	0.021	0.045	0.020	0.0050
		10 550	5640	0.091	1.10	−0.100	0.213[a]
Drawn 84 per cent	400	3 040	4500	0.022	0.059	0.047	0.0029
		15 000	5750	0.111	0.324	0.161	0.028
	500	320	5100	0.003	0.110	0.025	0.0164
		530	5640	0.005	0.280	0.090	0.0336
		1 820	5750	0.021	2.49	−0.243	0.47[a]
		4 960	830	0.055	2.63	−1.52	5.0[a]

TABLE III.—*Concluded.*

Temper	Testing Temperature, deg. Fahr.	Stress, psi.	Total Time, hr.	Initial Extension, per cent	Total Creep, per cent	Creep	
						Intercept, e_0, per cent	Rate, v, per cent per 1000 hr.
Cu-Ni-P Alloy							
Quenched from 1450 F. and Aged 4 hr. at 850 F.	300	10 250	3700	0.062	0.011	0.011	<0.0001
		20 200	5150	0.110	0.021	0.019	0.0004
		30 400	5850	0.175	0.036	0.028	0.0014
		40 200	5900	0.255	0.092	0.077	0.0025
		45 600	4030[c]	0.320	0.221[c]	0.127	0.023
	400	7 200	5400	0.056	0.020	0.018	0.0003
		10 050	5050	0.065	0.030	0.024	0.0011
		15 250	1600[c]	0.100	0.026[c]	0.012	0.015
	500	3 050	5100	0.019	0.012	0.009	0.0006
		5 100	5100	0.033	0.029	0.017	0.0024
		5 900	5400	0.036	0.043	0.018	0.0046
		8 100	5250	0.050	0.048	0.011	0.0071
		10 150	5750	0.065	0.294	−0.062	0.062[a]
		20 500	50[c]	0.150	0.038[c]	...	...
Quenched from 1450 F. Aged 4 hr. at 850 F. and Drawn 84 per cent	300	10 250	5150	0.070	0.012	0.012	<0.0001
		25 050	5150	0.175	0.021	0.019	0.0003
		39 700	5150	0.260	0.030	0.026	0.0008
		50 000	5900	0.316	0.059	0.052	0.0012
		59 700	5900	0.377	0.077	0.067	0.0017
		69 900	5900	0.460	0.149	0.124	0.0042
	400	10 050	5400	0.063	0.018	0.018	<0.0001
		21 100	5400	0.133	0.041	0.038	0.0006
		30 500	5100	0.188	0.039	0.033	0.0013
		39 800	5100	0.245	0.066	0.051	0.0029
	500	10 250	5100	0.052	0.033	0.028	0.0010
		15 350	5000	0.082	0.059	0.054	0.0009
		24 300	5100	0.131	0.100	0.075	0.0049
		35 000	5400	0.200	0.236	0.156	0.015
		49 200	3100	0.295	0.670	0.240	0.138
		49 200	4130	0.295	0.825	0.125	0.17[d]
Cu-Ni-P-Te Alloy							
Quenched from 1450 F. and Aged 1.5 hr. at 850 F.	300	20 700	6000	0.130	0.030	0.029	0.0002
		30 900	6000	0.200	0.053	0.043	0.0017
		35 000	6000	0.228	0.101	0.080	0.0035
		41 500	6000	0.300	0.274	0.204	0.012
	400	10 000	6000	0.065	0.039	0.030	0.0016
		15 050	6000	0.098	0.074	0.048	0.0043
		20 150	6000	0.132	0.103	0.062	0.0070
		29 500	3360	0.195	0.260	0.095	0.049
		29 500	6000	0.195	0.415	−0.402	0.136[e]
	500	2 050	6000	0.013	0.011	0.005	0.0010
		5 050	6000	0.032	0.086	0.039	0.0078
		10 150	6000	0.065	0.351	0.057	0.049
Quenched from 1450 F., Drawn 30 per cent and Aged 1.5 hr. at 770 F.	300	19 900	6000	0.121	0.020	0.017	0.00045
		29 900	6000	0.185	0.050	0.041	0.0015
		39 800	6000	0.250	0.079	0.064	0.0025
		48 800	6000	0.310	0.128	0.089	0.0064
		59 800	5800	0.425	0.640	−0.408	0.18[f]
	400	10 000	6000	0.061	0.046	0.029	0.0028
		15 000	6000	0.091	0.097	0.069	0.0047
		20 400	6000	0.125	0.145	0.095	0.0083
		30 100	6000	0.184	0.351	0.088	0.044[a]
		39 900	600	0.245	0.280	0.095	0.31[g]
		39 900	900	0.245	0.383	...	...
	500	3 050	6000	0.021	0.044	0.039	0.0008
		5 950	6150	0.040	0.068	0.040	0.0045
		10 200	6860	0.070	0.195	0.080	0.017
		15 000	5050	0.100	0.682	−0.040	0.143[h]

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[d] Third stage of creep, accelerating creep rate, terminating in brittle failure at 4400 hr.

[e] Third stage of creep, accelerating creep rate, terminating in brittle failure at 6100 hr.

[f] Third stage of creep, accelerating creep rate, terminating in brittle failure at 5800 hr.

[g] This value applies to second stage of creep at indicated time. Test entered third stage of creep thereafter and specimen failed with a brittle fracture shortly after maximum time indicated for test.

[h] Third stage of creep, accelerating creep rate, terminating in brittle failure at 5050 hr.

TABLE IV.—SUMMARY OF CREEP PROPERTIES.

Material	Temper	Temperature, deg. Fahr.	Stress for Designated Creep Rate, psi.		
			0.001 per cent per 1000 hr.	0.01 per cent per 1000 hr.	0.1 per cent per 1000 hr.
Electrolytic copper	Annealed, 0.025 mm.	300	1 100[a]	2 950	8 000
		400	400[a]	1 300	4 000
		500	100[a]	350	1 150
	Drawn 84 per cent	300	5 000[a]	9 400	14 700[b]
		400	1 000	2 100	4 350
Oxygen-Free copper	Annealed, 0.025 mm.	300	1 600	3 600	8 200
		400	500[a]	1 500	4 800
		500	100[a]	400	1 700
	Drawn 84 per cent	300	...	8 300	12 800[b]
		400	650[a]	1 750	5 000
Deoxidized copper	Annealed, 0.013 mm.	300	2 900	7 200	10 000[b]
		400	500[a]	2 100	5 400
		500	250[a]	700	1 950
	Annealed, 0.013 mm. and stretched 1 one per cent	300	2 600[a]	5 600	12 000[a]
		400	1 000[a]	2 500	5 600[a]
		500	250[a]	750[a]	2 100
	Annealed, 0.013 mm. and stretched 6 per cent	300	...	14 000	19 000
		400	1 150[a]	2 900	7 500
		500	250	900	3 300
Arsenical copper	Annealed, 0.045 mm.	300	4 000[a]	8 400	>10 000
		400	2 200	5 200	>7 000
		500	1 250	2 600	5 500
	Drawn 84 per cent	300	26 000	40 000	46 000
		400	12 800	27 500	35 500
		500	1 500	2 000[b]	3 750[b]
Red brass	Annealed, 0.060 mm.	300	5 200	8 600	>10 000
		400	1 250	5 500	9 000
		500	...	2 250	5 200
	Drawn 37 per cent	300	7 800	22 000	>30 000
		400	2 800	10 000	18 700
		500	...	850	3 300
	Drawn 84 per cent	300	12 000	37 000	>55 000
		400	1 000	2 600	6 800[a]
		500	...	600	1 450
Admiralty	Annealed, 0.055 mm.	300	13 200	19 500[a]	...
		400	3 100	11 000	>13 000
		500	950	2 150	4 600[b]
	Annealed, 0.018 mm.	300	5 400	19 000	25 500[a]
		400	...	2 900	9 600
		500	...	300[a]	1 350
	Drawn 60 per cent	300	10 000	32 000	52 000[a]
		400	...	1 500[b]	9 600[b]
		500	150[a]	380	1 050
Aluminum brass	Annealed, 0.030 mm.	300	5 900	13 400	26 500
		400	900[a]	3 200	8 400
		500	200[a]	650	2 000[a]
	Annealed, 0.015 mm.	300	6 700	14 300	26 500
		400	900[a]	2 900	7 700
		500	...	600[a]	1 750[a]
	Drawn 37 per cent	300	10 000[a]	35 000	54 000[a]
		400	9 000[a]	21 000	30 500
		500	...	1 000	2 400
Naval brass	Annealed at 1000 F.	300	4 800	12 000	>20 000[b]
		400	600[a]	1 900	5 000
		500	...	250[a]	750
	Drawn 37 per cent	300	1 550	12 400	25 000
		400	...	3 000	9 000
		500	...	100[a]	400

[a] By extrapolation.
[b] Produces accelerating creep rate.

TABLE IV.—*Continued.*

Material	Temper	Temperature, deg. Fahr.	Stress for Designated Creep Rate, psi.		
			0.001 per cent per 1000 hr.	0.01 per cent per 1000 hr.	0.1 per cent per 1000 hr.
3 per cent silicon bronze	Annealed, 0.100 mm.	300	9 900	14 800	>17 000
		400	5 900	8 500	>11 000
		500	3 500	5 300	8 000
	Annealed, 0.008 mm.	300	10 400	23 000	29 000
		400	1 600^a	5 000	15 000
		500	700^a	1 700	4 200
	Drawn 37 per cent	300	19 000^a	40 000	>60 000
		400	4 500	24 000	>30 000
		500	...	2 500	7 500^b
Phosphor bronze, type A	Annealed, 0.050 mm.	300	11 000	17 000	...
		400	3 700	>10 000	...
		500	1 150	3 600	9 000
	Drawn 84 per cent	300	5 000	34 000	>50 000
		400	1 500^a	7 500	>15 000
		500	...	200^a	1 100
Cu-Ni-P alloy	Quenched from 1450 F. and aged 4 hr. at 850 F.	300	28 000	>40 000	...
		400	10 000	14 500	>15 000
		500	3 700	8 200	>10 000^b
	Quenched from 1450 F., aged 4 hr. at 850 F. and drawn 84 per cent	300	46 000	>70 000	...
		400	27 000	>40 000	...
		500	13 000	31 000	47 000^c
Cu-Ni-P-Te alloy	Quenched from 1450 F. and aged 1.5 hr. at 850 F.	300	29 000	40 000	...
		400	8 000^a	21 000	>30 000^c
		500	2 000	5 600	>10 000
	Quenched from 1450 F., drawn 30 per cent and aged 1.5 hr. at 770 F.	300	27 000	52 000	59 000
		400	...	22 000	35 000^c
		500	3 400	8 300	14 500

[c] Produces accelerating creep rate and early failure.

hr. is 0.088 at 6000 and 0.072 at 11,000 hr. The other test is for a stress of 39,900 psi. at 300 F. on aluminum brass as drawn 37 per cent, and its creep rate is 0.016 at 6000 and 0.010 at 11,000 hr. These values illustrate the magnitude of the changes in creep rate which occur between the indicated times. Although the changes in both cases are quite small when considered on the basis of absolute values, the change in the first case is relatively small, whereas the change in the second case is relatively large. The latter demonstrates the importance of using a fixed time in evaluating creep rates which are to serve as a basis of comparison of various materials under one or more conditions of exposure. Obviously the degree of freedom which can be exercised in this connection is determined by the rate of change of creep rate with time. Wherever possible in the present work, only those creep rates at about 5000 to 6000 hr. are used for comparative purposes.

The Coppers:

The electrolytic, oxygen-free, and arsenical coppers were tested in tempers as annealed and as drawn 84 per cent. The grain sizes for the annealed conditions are given in Table I. The creep characteristics of the deoxidized copper which have been determined and reported previously (2) are introduced briefly here for purposes of comparison with the properties of the other types of copper. This copper has also been used in some tests to determine the effect of small amounts of cold working on the creep properties of the annealed material, where the cold working is effected by stretching 1 and 6 per cent at room temperature prior to the creep tests.

The initial tensile strength and elongation values of the four types of copper in the annealed condition are virtually the same even though there is some difference in their grain sizes. The same is true for the hard-drawn temper except in the case of arsenical copper, the tensile strength of which is about 10 per cent greater than that of the other hard-drawn coppers.

Consideration of the effect of exposure in the creep tests on the microstructure and tensile properties of the different types of copper is important in the appraisal of their creep properties. Annealed specimens which were stressed in the creep tests and others which were exposed to the same temperatures but were not stressed showed essentially no change in tensile properties as a result of the prolonged exposures. For the sake of brevity, the test values demonstrating this behavior are not included in Table II.

The hard-drawn electrolytic copper became about half recrystallized during the course of the tests at 300 F. and was completely recrystallized at 400 F. very shortly after the creep loads were applied. The tensile properties listed in Table II demonstrate these points. Interestingly enough, the hard-drawn oxygen-free copper parallels electrolytic copper very closely in recrystallization and softening characteristics. The hard-drawn arsenical copper is much more stable than these, for there was no significant softening and no apparent change in microstructure at 300 F. At 400 F. it softened slightly but there was still no indication of any recrystallization after the long exposure. Only at 500 F. did this type of copper become completely recrystallized and then only after a long period of time. The hard-drawn deoxidized copper is intermediate to the arsenical copper and the electrolytic and oxygen-free coppers in respect to recrystallization and softening characteristics. As reported previously (2), it softened slightly after long exposure at 300 F. At 400 F. it softened considerably and became somewhat more than half recrystallized in the course of creep tests which were of about 6000-hr. duration. It was almost completely recrystallized and softened after having been exposed only 24 hr. at 500 F.

The stretching of the annealed, deoxidized copper by 1 and 6 per cent produced some increase in tensile strength as shown in Table I. The tensile properties and microstructure of the material which was stretched 1 per cent were unaffected by exposures in the creep tests at 300, 400, and 500 F. The material which was stretched 6 per cent retained its tensile properties after creep-test exposures at 300 F. only. Slight softening occurred at 400 and somewhat more at 500 F. This is shown in Table II by the lower values of tensile strength and higher values of elongation after exposures at these higher temperatures. The microstructure of the material, stretched 6 per cent, was apparently unchanged after exposures at 300 and 400 F., but at 500 F. recrystallization occurred at the surface to a depth of about 0.008 in., and also at a few random points throughout the cross-section of the wire specimens. The grain size in the recrystallized areas was about 0.100 mm. The greater part of the metal did not recrystallize, however, but retained the original fine grain structure. In the absence of any visible recrystallization, the softening effected by exposures at 400 F. may be attributed to recovery, whereas at 500 F. the softening is produced by both recovery and recrystallization.

The relative positions of the curves in Fig. 1 relating stress and creep rate of electrolytic copper at 300 F. show that the hard-drawn electrolytic copper is

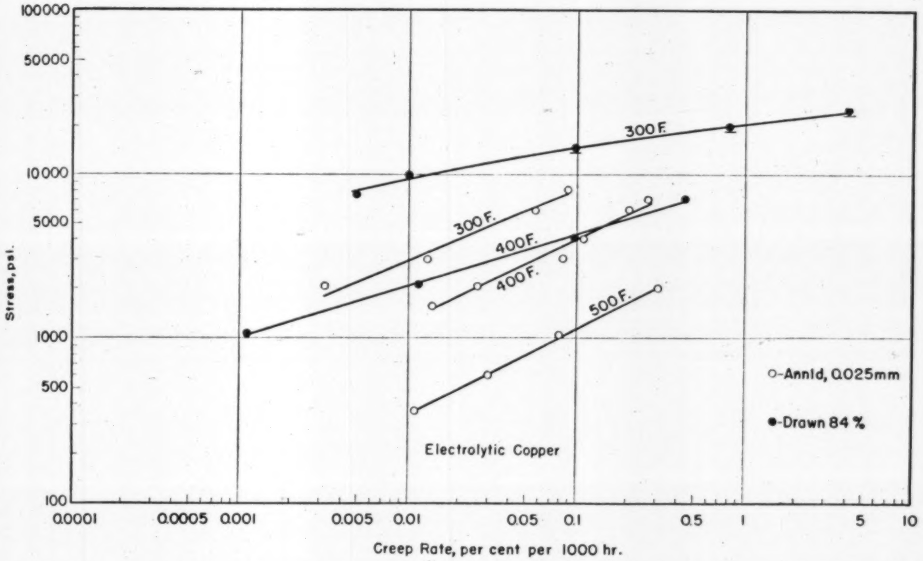

FIG. 1.—Stress and Creep Rate Relationships (Log Scales) at 300, 400, and 500 F. for Annealed and Hard-Drawn Electrolytic Copper.

"0.025 mm." indicates the grain size of the annealed material. Similar designations are used in other figures.

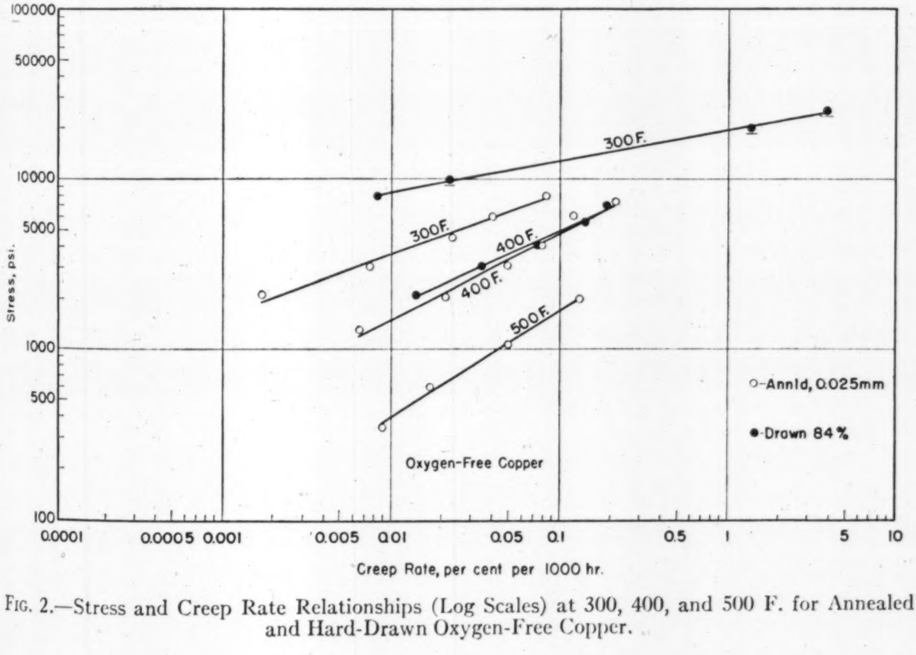

FIG. 2.—Stress and Creep Rate Relationships (Log Scales) at 300, 400, and 500 F. for Annealed and Hard-Drawn Oxygen-Free Copper.

definitely more resistant to creep at 300 F. than is the annealed material. Attention is called to the fact that the hard-drawn copper had entered the third stage of creep, that of accelerating rate, for stresses of about 15,000 psi. and higher at this temperature. At 400 F. there is much less difference in the creep characteristics of the two tempers as shown by the close proximity of the stress-creep rate curves. This similarity results from the complete recrystallization and softening of the hard-drawn material before the application of the creep-test loads and the subsequent behavior of the copper as an annealed material. This complete softening of the hard drawn copper at 400 F. obviated its being tested at any higher temperature and therefore only the annealed temper was tested at 500 F.

The stress-creep rate relationships shown in Fig. 2 for the oxygen-free copper are very similar to those for electrolytic copper. This might be expected in view of the similarity in softening characteristics which have already been described. Creep properties are in general dependent on composition, and in the case of these two types of copper which have nearly the same compositions, the consideration of purity becomes very important. The annealed electrolytic and oxygen-free coppers have electrical conductivities of 101.0 and 101.4 per cent IACS, respectively. The impurities in the oxygen-free copper must be exceedingly low to permit this high conductivity, and because they are present in such very small quantities, the impurities have little effect on creep characteristics. The most significant difference between these two types of copper is with regard to oxygen content. The electrolytic copper contains about 0.04 per cent oxygen, most of which is present in the separate phase, cuprous oxide. The copper matrix, which comprises the mass of this oxygen-bearing copper, must have very nearly the same composition as the oxygen-free copper in view of the virtual identity of their conductivity values.

The superior creep resistance of the hard-drawn arsenical copper at 300 and 400 F. is clearly demonstrated in Fig. 3 where the stress-creep rate curves for this material at these temperatures lie far above the corresponding curves for the annealed temper. At 500 F. the creep strength for the hard-drawn temper is greatly diminished, and for tests at stresses of 2000 psi. and higher the plotted points on the stress-creep rate curve indicate that those tests had entered the stage of accelerating creep rate. These findings can be explained in terms of recrystallization and softening. It has already been pointed out that no recrystallization and little or no softening of the hard-drawn arsenical copper occurred at 300 and 400 F. This stability is manifest in high creep strength at these temperatures. At 500 F., however, the material recrystallized and softened throughout the course of the creep tests. This instability lowers the creep resistance of the hard-drawn material to such an extent that it is inferior to the initially annealed temper over most of the range of stresses which were investigated at this temperature.

The relationships between stress required to produce a creep rate of 0.01 per cent per 1000 hr. and temperature from 300 to 500 F. are plotted in Fig. 4 so that a ready comparison of the creep strengths of these four types of copper is provided. For both annealed and hard-drawn conditions the curves for the electrolytic and oxygen-free coppers are located close together and at positions which indicate the lowest creep strength of the four types of copper. The curves for the deoxidized copper are

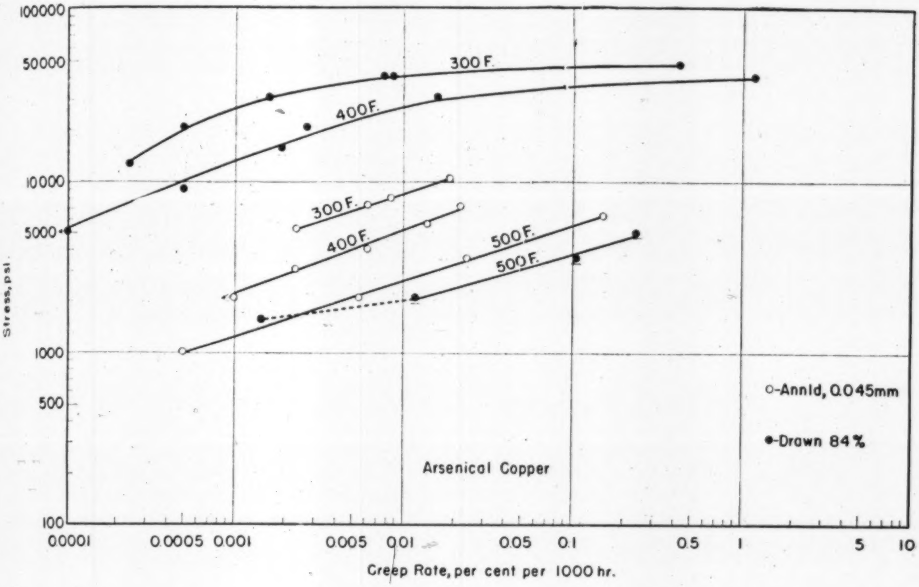

FIG. 3.—Stress and Creep Rate Relationships (Log Scales) at 300, 400, and 500 F. for Annealed and Hard-Drawn Arsenical Copper.

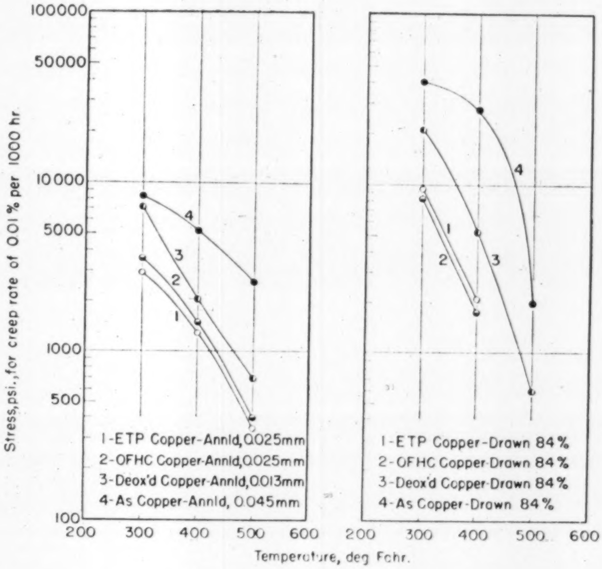

FIG. 4.—Creep Strength (Log Scale) and Temperature Relationships for Electrolytic Copper, Oxygen-Free Copper, Deoxidized Copper, and Arsenical Copper.

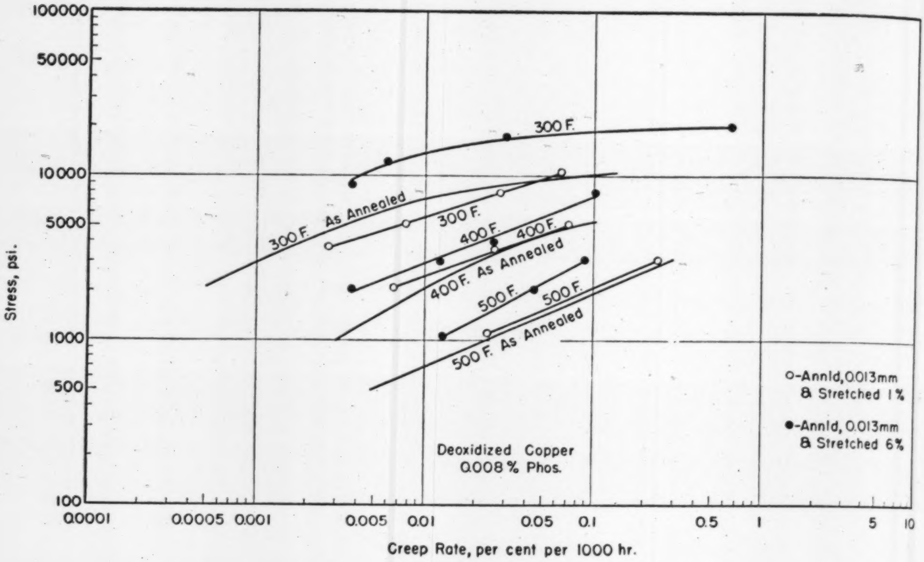

FIG. 5.—Stress and Creep Rate Relationships (Log Scales) at 300, 400, and 500 F. for Annealed and Hard-Drawn Deoxidized Copper.

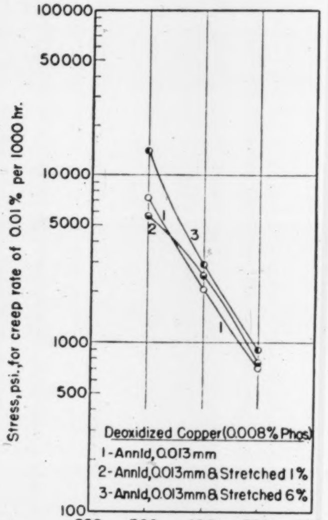

FIG. 6.—Creep Strength (Log Scale) and Temperature Relationships for Deoxidized Copper.

next in order of increasing creep strength and their relative positions indicate considerable superiority of this type of copper over the first two. The topmost curve in each temper is for the arsenical copper, which has the highest creep strength.

The creep tests which were made on the deoxidized copper as stretched 1 and 6 per cent after the final anneal are well summarized in Fig. 5, which shows the relationships between stress and creep rate for this copper as annealed and as stretched, and in Fig. 6, which shows the relationships between creep strength and temperature for the same conditions. The effect of the 1 per cent stretching seems quite insignificant at all three test temperatures, but the 6 per cent stretching produces a definite increase in creep strength, particularly at 300 F. As in the case of severely cold-worked conditions, the creep characteristics of these lightly worked materials are functions of their initial properties and their stabilities at elevated temperatures. The magnitude of the effect of prestretching 1 per cent is so small that the material behaves very much like the as-annealed material in every respect.

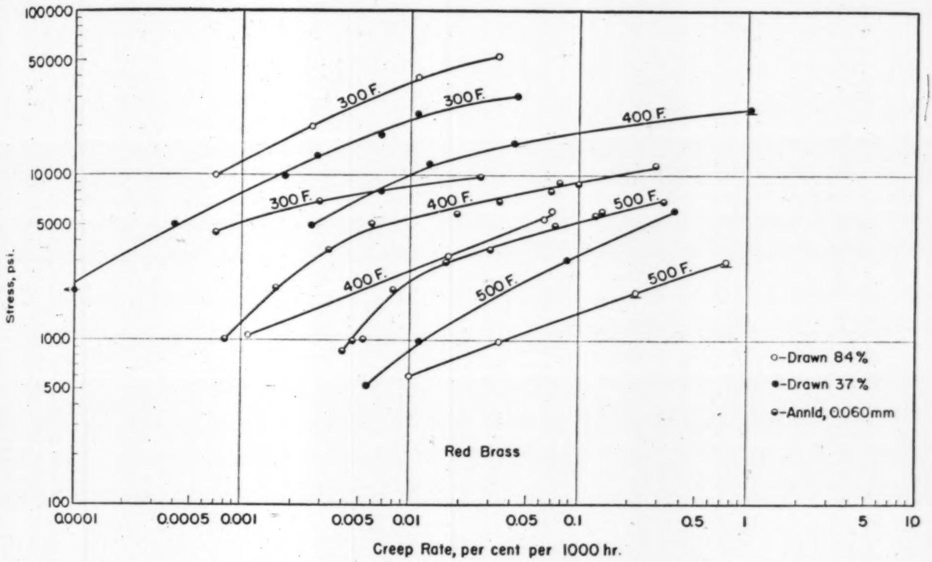

FIG. 7.—Stress and Creep Rate Relationships (Log Scales) at 300, 400, and 500 F. for Annealed and Hard-Drawn Red Brass.

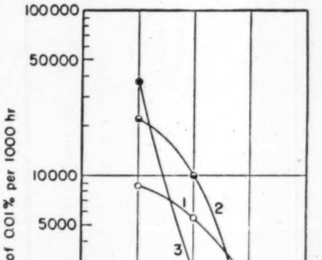

FIG. 8.—Creep Strength (Log Scale) and Temperature Relationships for Red Brass.

Prestretching 6 per cent, on the other hand, has a definite hardening effect which is manifest in higher tensile strength and greater resistance to creep, particularly at the lower temperatures. At higher temperatures, such as 500 F., this material recrystallizes and softens to some extent and its creep strength is correspondingly lowered.

Red Brass:

The curves relating stress, creep rate, and temperature for red brass are shown in Figs. 7 and 8. The previously reported data (1) for this alloy were considerably less extensive. While most of the new information consists of test results for stresses which supplement conditions previously discussed, the results for the annealed material at 500 F. are entirely new. The annealed material is shown to be superior to the two drawn tempers at 500 F.

Admiralty:

Admiralty was tested in two annealed tempers, 0.055 and 0.018-mm. grain size, and as drawn 60 per cent. The stress-creep rate relationships are shown in Fig. 9 and the creep strength-tempera-

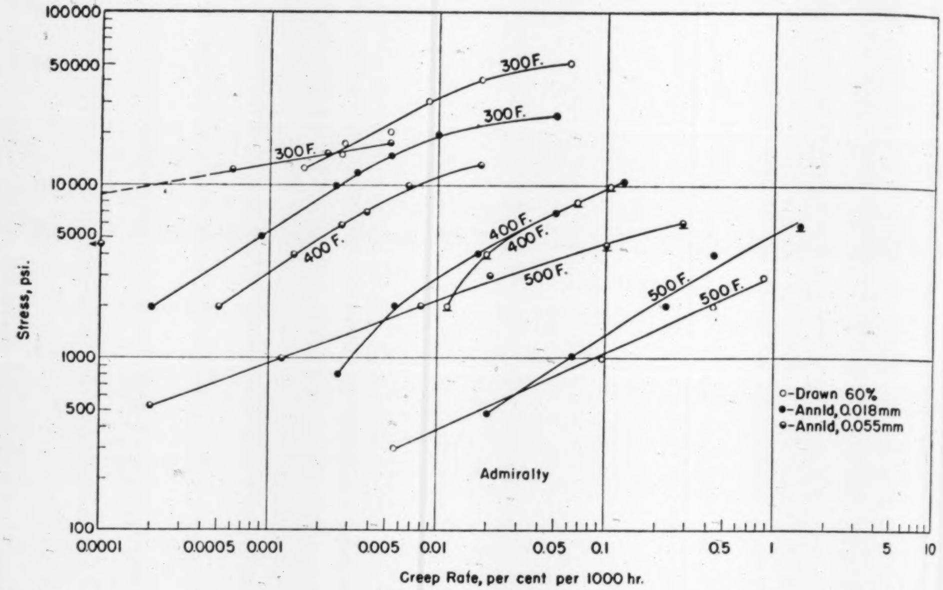

FIG. 9.—Stress and Creep Rate Relationships (Log Scales) at 300, 400, and 500 F. for Annealed and Hard-Drawn Admiralty.

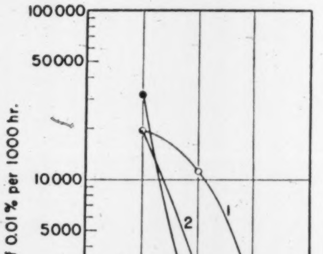

FIG. 10.—Creep Strength (Log Scale) and Temperature Relationships for Admiralty.

ture relationships in Fig. 10. Only the data on creep at 400 F. of the material with 0.055-mm. grain size have previously been reported in any great detail.

The creep characteristics of this alloy in the annealed condition are dependent on grain size. Except for the range of higher stress at 300 F., the material of larger grain size has appreciably greater creep strength at 300, 400, and 500 F.

The relative creep strength for the hard-drawn temper as compared with the annealed tempers changes with temperature. At 300 F. the drawn temper generally has the greatest resistance to creep. The microstructure and tensile properties of the drawn material are unaffected at this temperature. At 400 F., however, the material recrystallizes slowly and is about 75 per cent recrystallized after exposure for 6000 hr. The microstructure at 400 F. changes continuously because of this progressive recrystallization throughout the duration of the creep tests and, as might be expected, the resulting creep shows evidence of this structural instability. A stage of accelerating creep rate develops after

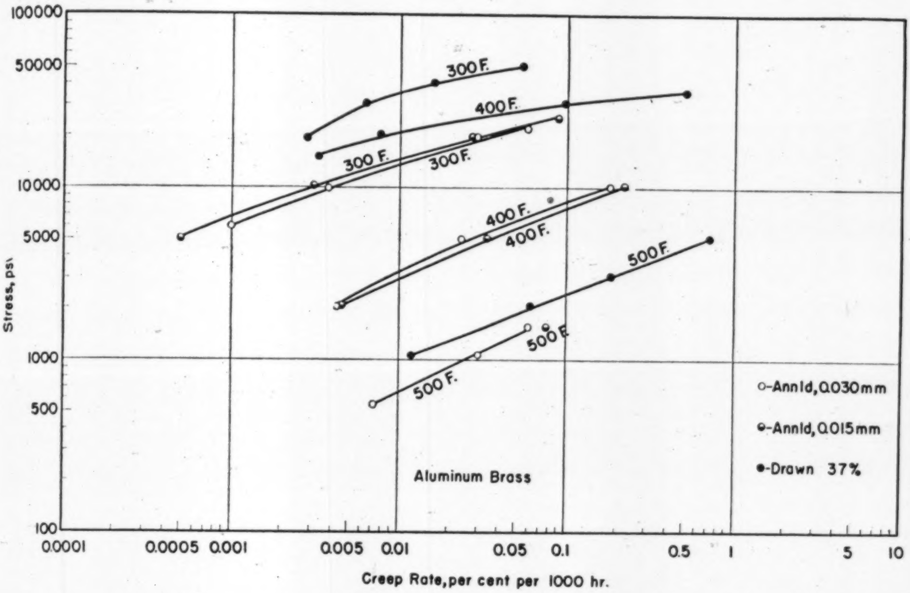

FIG. 11.—Stress and Creep Rate Relationships (Log Scales) at 300, 400, and 500 F. for Annealed and Hard-Drawn Aluminum Brass.

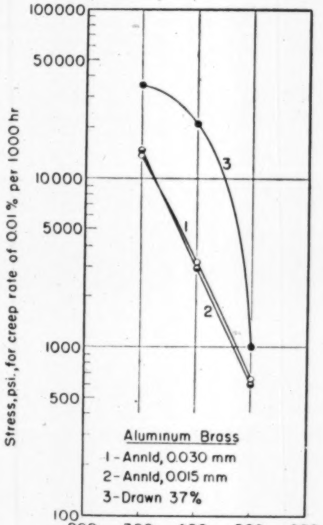

FIG. 12.—Creep Strength (Log Scale) and Temperature Relationships for Aluminum Brass.

approximately 2000 hr. It is likely that creep beyond the 6000-hr. test period would gradually diminish in rate after the structure becomes completely recrystallized. This expected behavior would be very similar to that which was found in creep tests at 400 F. on 70–30 brass, drawn 37 per cent (1), where a stage of decreasing creep rate followed the stage of accelerating creep rate. The close association between creep and recrystallization characteristics is illustrated in both instances. The near coincidence of the 400 F. stress-creep rate curve for the drawn temper of admiralty with the corresponding curve for the annealed material of 0.018 mm. grain size shows that the creep rate near the end of the tests on the drawn material are indicative of its long-term creep characteristics and that they do not actually represent the commonly regarded third stage of creep which leads to failure.

At 500 F. the drawn temper has about the same creep strength as the annealed material with 0.018-mm. grain size. Since the drawn material recrystallized completely at 500 F. very shortly after the beginning of the tests, its creep behavior was characteristic of an annealed material rather than a drawn one and the recrystallized grain size probably was the primary factor determining its creep strength. The relative position of the stress-creep rate curve for this material at 500 F. in Fig. 9 is largely consistent with the grain size of about 0.010 mm. developed by recrystallization during the creep tests.

Aluminum Brass:

Aluminum brass was tested in two annealed tempers with grain sizes of 0.030 and 0.015 mm., respectively, and as drawn 37 per cent. The various curves which describe the creep characteristics of these materials are shown in Figs. 11 and 12. The microstructure and tensile properties of the annealed materials were not significantly affected by the exposures in the creep tests. The small differences in creep strength of the two annealed tempers are consistent with the small differences in their grain sizes. The relative magnitudes of their creep strengths at different temperatures are of interest, however, since they indirectly indicate the equicohesive temperature of this alloy. At 300 F. the material of smaller grain has somewhat greater creep strength than the material of larger grain size, whereas at 400 and 500 F. the reverse is true. This is illustrated by the crossing of curves 1 and 2 in Fig. 12. The temperature, approximately 330 F., at the point of crossing may be considered as the equicohesive temperature since the creep strength at that temperature is independent of grain size, at least for the two grain sizes used.

The aluminum brass in the drawn temper has greater creep strength than in the annealed tempers at all three temperatures. At the two lower temperatures, 300 and 400 F., the greater creep strength of the drawn material is associated with its structural stability and retention of physical properties The margin of its superiority over the annealed tempers at these temperatures is large, about 2.5 to 1 at 300 F. and about 7 to 1 at 400 F. At 500 F., however, the drawn material is unstable and recrystallizes. The rate of its recrystallization is relatively low and about half of the creep test period of 6000 hr. is required for complete recrystallization. This effects a considerable lowering of its resistance to creep. The grain size of the drawn material after recrystallization at 500 F. is about 0.020 mm., and one might therefore expect that the stress-creep rate curve for the drawn material at 500 F. in Fig. 11 should be located between the corresponding curves for the initially annealed materials of 0.015 and 0.030-mm. grain size. A possible reason that it is not so located is that the material recrystallizes slowly during a large portion of the creep test period and therefore retains during a significant part of the test at least some of the higher resistance to creep which is characteristic of the drawn material.

The microstructure and tensile properties of the drawn material after long exposure at 500 F. are similar to those of the annealed materials and its creep strength is only slightly higher. The tests on the drawn material at 500 F. provide an indication of the complex effect of prolonged recrystallization on the course of creep. In these tests the creep-time curves progressed through two major stages of creep: first, one of accelerating creep rate and, thereafter, one of slowly decreasing creep rate which was still in progress when the tests were terminated. The first major stage of creep may have been preceded by one of decreasing creep rate for a very short period of time, perhaps a few hours. Because of its extremely short duration, this preliminary stage is not considered to be significant. For the various stresses the first major stage of creep lasted for periods of time ranging from about 300 to about 1200 hr., after which the second major stage prevailed. The creep rates listed in Table III for these tests refer to this last stage.

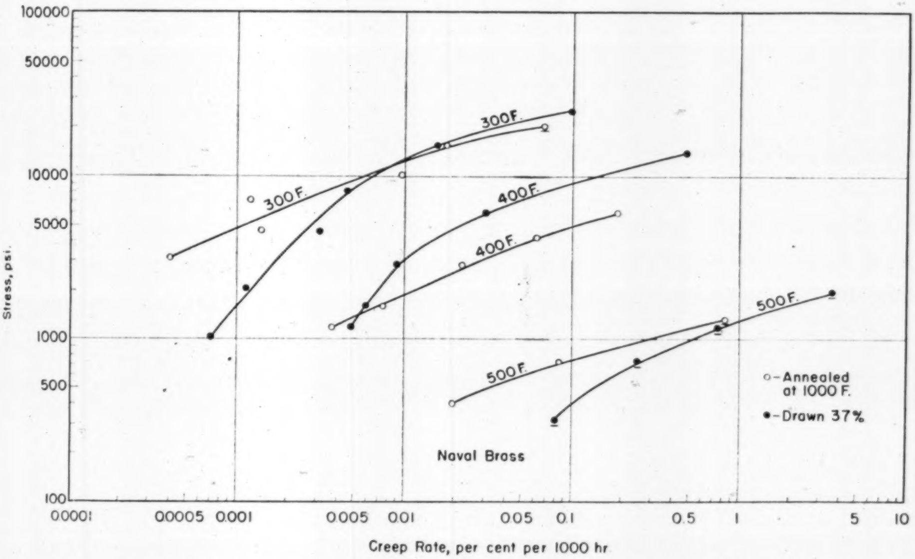

FIG. 13.—Stress and Creep Rate Relationships (Log Scales) at 300, 400, and 500 F. for Annealed and Hard-Drawn Naval Brass.

The creep properties at 300, 400, and 500 F. of admiralty and aluminum brass in annealed tempers are very similar, and they are also about the same as those of 70–30 brass (1). The stress required to produce a creep rate of 0.01 per cent per 1000 hr. at 400 F. in 70–30 brass, admiralty, and aluminum brass as annealed with grain sizes of 0.016, 0.018, and 0.015 mm., respectively, is 2900 psi. in each instance. At other temperatures and for different creep rates there are actual differences in the creep strengths of these three materials but the differences are relatively small. It is apparent, therefore, that the tin and aluminum in these special brasses contribute little to creep strength in annealed tempers.

In drawn tempers there are some fairly significant differences in the creep properties of these three alloys. The differences stem largely from the different recrystallization characteristics of the three alloys. The presence of aluminum in aluminum brass as drawn 37 per cent imparts a degree of structural stability sufficient to restrict recrystallization completely at 400 F. and to retard it at 500 F. On the other hand, the presence of tin in admiralty does not appear to have any significant effect upon recrystallization characteristics. In accordance with the relatively greater stability of aluminum brass at 400 and 500 F. with regard to softening, the creep strength of this alloy is higher than that of admiralty and 70–30 brass in drawn tempers at these temperatures. The differences are rela-

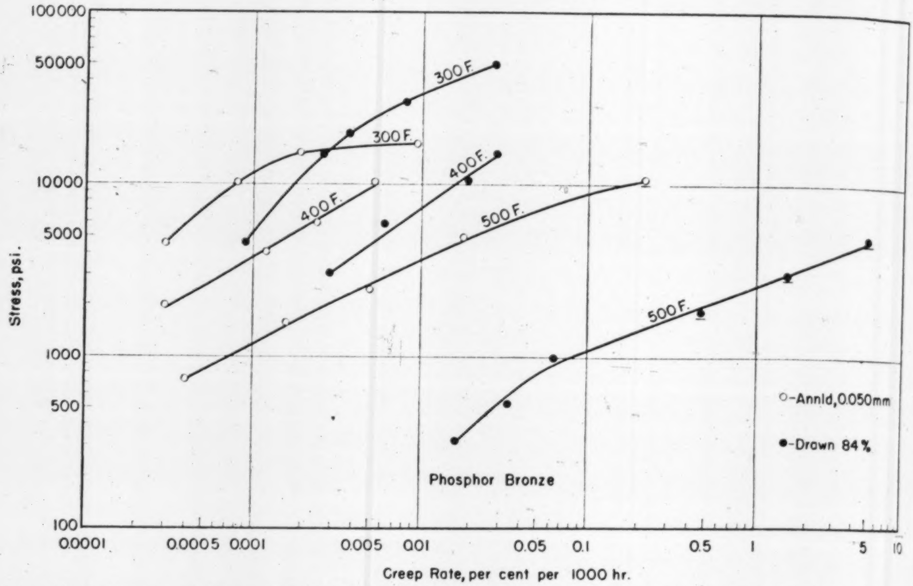

FIG. 14.—Stress and Creep Rate Relationships (Log Scales) at 300, 400, and 500 F. for Annealed and Hard-Drawn Phosphor Bronze.

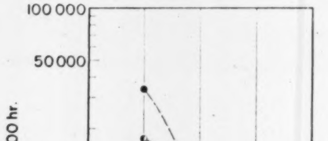

FIG. 15.—Creep Strength (Log Scale) and Temperature Relationships for Naval Brass and Phosphor Bronze.

tively greater as the temperature increases up to those temperatures where all three of the alloys recrystallize. The temperatures at which these differences largely disappear depend on the degree of reduction of the initially drawn tempers.

Naval Brass:

The earlier work (1) on naval brass is now amplified by additional test results, particularly at 300 and 500 F. Figures 13 and 15 summarize the results at all three test temperatures. Referring to Fig. 13, it will be noted that the creep rates for the two tempers of this alloy at 300 F. are nearly equal for stresses in the vicinity of 11,000 psi. For stresses less than this the annealed material has the greater creep resistance, as shown by the divergence of the two stress-creep rate curves in that region and the higher position of the curve for the annealed material. For stresses above approximately 11,000 psi., the drawn material has the greater creep resistance. At 20,000 psi., the annealed metal had entered a stage of accelerating creep rate while the drawn material stressed at 24,900 psi. still extended at a decreasing rate at the end of the test period.

The stress-creep rate curve for the drawn temper of this alloy at 500 F., which was not included in the previous paper (1), is now shown in Fig. 13. Each of the plotted points for this curve represents an accelerating rate of creep. The value of stress required to produce a rate of 0.01 per cent per 1000 hr. for this material at 500 F. should be considered as only approximate, for considerable extrapolation was required to obtain it. Furthermore, the fact that the points of higher stress represented accelerating creep rates lends uncertainty to the exactness of the value. However, it is clearly indicated that the stress which would produce this creep rate is very low.

3 per cent Silicon Bronze:

A few additional creep data for 3 per cent silicon bronze at 300 F. are included in Table III to supplement the previously published work (1) on this alloy. The general conclusions regarding the creep characteristics of the alloy are not altered by this additional information.

Phosphor Bronze:

The stress-creep rate and temperature relationships included in the earlier work (1) for the phosphor bronze are now more completely defined in Figs. 14 and 15. At 300 F. the alloy as annealed is distinctly superior to that as drawn for stresses below about 15,000 psi., although the creep rates are very low for both tempers in this stress range. For higher stresses, the trend with respect to temper is reversed and the superiority of the drawn material is very marked for creep rates in excess of 0.005 per cent per 1000 hr. The flattening of the stress-creep rate curve for the annealed temper in the latter range is undoubtedly associated with the yield strength of the material.

For stresses up to at least 10,000 psi. at 400 F., the annealed material is definitely superior to that as hard-drawn. For higher stresses it is likely that the alloy in the annealed condition would again show, as at 300 F., that stressing near or in excess of the yield strength would result in disproportionately greater creep. The alloy in the drawn temper showed no evidence of abnormally high creep at 15,000 psi. at this temperature.

Copper-Nickel-Phosphorus Alloy:

The copper-nickel-phosphorus alloy was tested in two tempers, (*a*) as quenched and aged, and (*b*) as quenched, aged and drawn 84 per cent. Creep properties for both conditions are shown graphically in Figs. 16 and 18. This age-hardenable alloy is characterized by very high resistance to creep at 300, 400, and 500 F. In general, the alloy has greater creep strength at the three temperatures than any of the other alloys which have been described in preceding sections. The creep properties for the heat-treated and hard-drawn temper are appreciably higher than those for the softer temper, which is heat-treated but not drawn. This is true even at 500 F. The excellent creep properties of the alloy must be regarded with some reservations, however, since both tempers are susceptible to intergranular separation at very high stresses. The separation is accompanied by a deterioration which is manifest in loss of tensile strength and ductility as shown in Table II. It will be observed, however, that this embrittlement occurs only under very severe conditions of exposure with regard to stress and temperature, that is, conditions which are more severe than those which might be encountered in normal service.

The tensile properties of the quenched and aged material are virtually unaffected by exposures at 300 F. with stresses up to 30,000 psi. The creep-test specimen which had been stressed at 40,200 psi. failed with a brittle fracture in the subsequent tension test at room temperature. When stressed at 45,600 psi. in the creep test at 300 F. the material failed with a brittle fracture after about 4000 hr. There was no evidence of embrittlement throughout the creep test specimens exposed at 400 F. at stresses up to 15,250 psi. except at the points where set screws attaching the extension rods had indented the test wires and produced some stress concentration. For the applied stress of 15,250 psi. this condition of stress concentration led to brittle fracture at one of the indentations after 1600 hr. At 500 F. there was no apparent embrittlement produced by a stress of 3050 psi., whereas it did occur at the next higher stress, 5100 psi. In those instances where the conditions of exposure are sufficiently severe to produce embrittlement, the time to failure depends on the actual values of stress and temperature. The combined effects of stress and temperature on the time to failure are illustrated by data for this material in Table III.

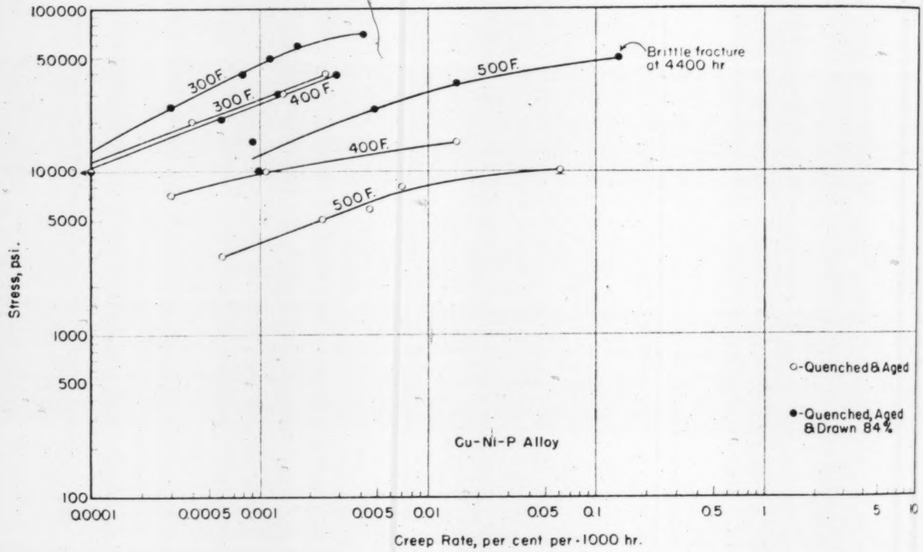

FIG. 16.—Stress and Creep Rate Relationships (Log Scales) at 300, 400, and 500 F. for Copper-Nickel-Phosphorus Alloy as Quenched and Aged, and as Quenched, Aged, and Drawn.

As quenched, aged, and drawn 84 per cent, the alloy resists embrittlement at considerably higher stresses than those enumerated for the quenched and aged condition. The maximum stresses which do not produce embrittlement for this condition are about 70,000, 40,000, and 35,000 psi. at 300, 400, and 500 F., respectively. The material is softened somewhat by long exposure at the elevated temperatures. Reductions in tensile strength after exposures in creep tests at stresses which do not exceed these limiting stresses range from about 3 to 12 per cent. Since

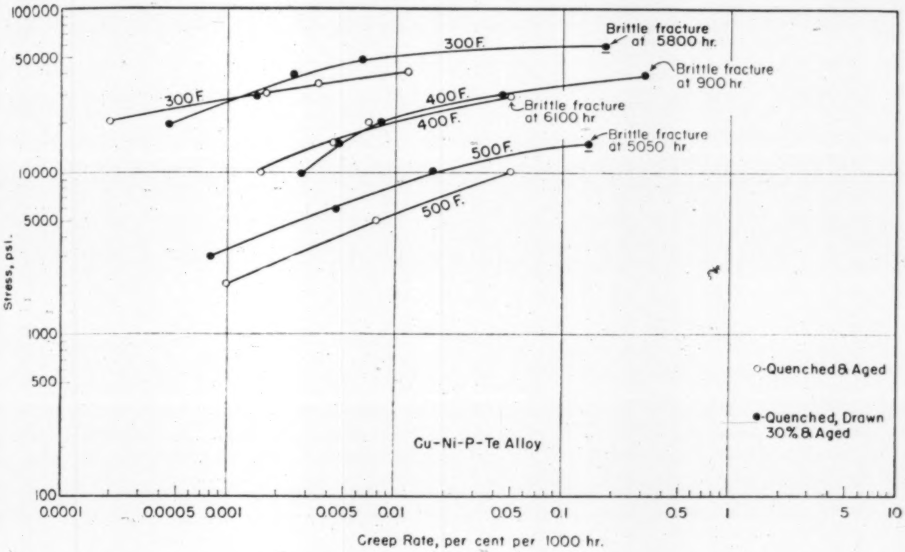

Fig. 17.—Stress and Creep Rate Relationships (Log Scales) at 300, 400, and 500 F. for Copper-Nickel-Phosphorus-Tellurium Alloy as Quenched and Aged, and as Quenched, Drawn, and Aged.

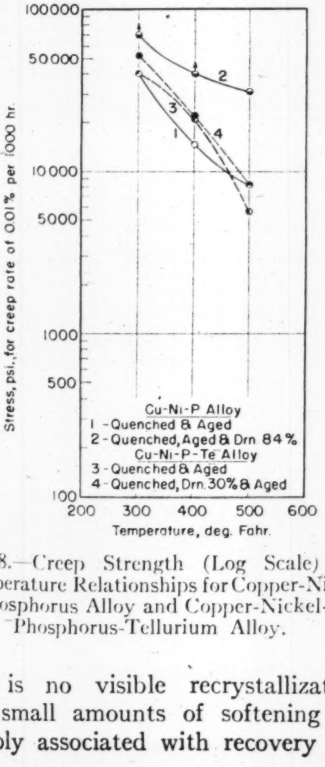

Fig. 18.—Creep Strength (Log Scale) and Temperature Relationships for Copper-Nickel-Phosphorus Alloy and Copper-Nickel-Phosphorus-Tellurium Alloy.

there is no visible recrystallization, these small amounts of softening are probably associated with recovery and slight overaging during the prolonged exposures at these temperatures.

Copper - Nickel - Phosphorus - Tellurium Alloy:

The copper-nickel-phosphorus-tellurium alloy was tested in two tempers, (*a*) as quenched from 1450 F. and aged 1.5 hr. at 850 F., and (*b*) as quenched from 1450 F., drawn 30 per cent and aged 1.5 hr. at 770 F. The stress, creep rate, and temperature relationships for these materials are shown in Figs. 17 and 18. Except for its tellurium content, which imparts free machining properties, this alloy is similar in composition to the copper-nickel-phosphorus alloy described in the previous section. In the quenched and aged condition the two alloys have about the same resistance to creep at 300, 400, and 500 F. This is evident in the proximity of curves 1 and 3 in Fig. 18. In the harder temper the tellurium-bearing alloy is heat-treated and drawn in a different sequence than the alloy without tellurium, and the degree of reduction is considerably smaller, 30 instead of 84 per cent. The tellurium-bearing alloy as quenched, drawn 30 per cent and aged, has lower tensile properties and less resistance to creep than the copper-nickel-phosphorus alloy as quenched, aged and drawn 84 per cent. In comparison with the other copper-base materials included in this paper, however, this material has very high creep strength.

The creep properties of the two tempers of the copper-nickel-phosphorus-tellurium alloy are about the same. Values of creep strength for given creep rates and temperatures are of the same order of magnitude for both tempers. The only significant difference occurs at 500 F., at which temperature the quenched, drawn, and aged material has the greater creep resistance over the entire range of stresses used in the creep tests.

The susceptibility of this alloy to intergranular separation is in general similar to that of the copper-nickel-phosphorus alloy, except for the fact that there is a much smaller quantitative difference between the two tempers of the tellurium-bearing alloy with regard to the stresses which produce intergranular separation. The time required to produce brittle failure in these materials at various stresses and temperatures is given in Table III and the magnitude of the effect on physical properties after exposure is given in Table II. Properties of the quenched and aged material after exposure at 500 F. indicate embrittlement even for unstressed specimens, whereas the material in the quenched, drawn, and aged temper appears to have been less affected. For exposures at 300 and 400 F. at stresses below those which produce intergranular separation and ultimate failure, there is no loss of tensile strength and ductility for either temper.

SUMMARY

The creep characteristics of several types of copper and of several copper alloys were determined at 300, 400, and 500 F. Of the various coppers, arsenical copper has the greatest creep strength. Deoxidized copper ranks second, and electrolytic copper and oxygen-free copper, which have very similar creep properties, rank third. This order of relative merit applies to both annealed and hard-drawn tempers. The creep properties of the hard-drawn coppers are closely associated with their individual softening characteristics. The marked superiority of the arsenical copper over the other coppers is largely derived from its much greater stability with regard to softening at elevated temperatures.

The effect of small amounts of cold work on creep properties of deoxidized copper was investigated. The creep resistance of the annealed material is improved somewhat by stretching 6 per cent but it is not significantly changed by stretching 1 per cent. The relative strengthening produced by the higher degree of reduction diminishes with increasing temperature of exposure.

The work on admiralty and aluminum brass showed that the creep characteristics for annealed tempers of these alloys are similar to those of 70-30 brass. The tin and aluminum in these special brasses contribute little to creep strength in annealed tempers. The creep strength for annealed conditions of both of these alloys generally increases with increasing grain size. There are minor exceptions to this, however, as for example in the case of aluminum brass at 300 F., where the smaller grain size has slightly greater creep strength. The creep properties of both alloys in drawn tempers are closely associated with their softening characteristics at the three elevated temperatures.

The age-hardenable copper-nickel-phosphorus and copper-nickel-phosphorus-tellurium alloys were found to have very high creep strength. Severe cold working of the copper-nickel-phosphorus alloy after heat treatment produced the greatest resistance to creep. Both alloys are susceptible to embrittlement for conditions of exposure which are severe with respect to stress and temperature.

The previously published work (1) on the creep characteristics of red brass, naval brass, 3 per cent silicon bronze and phosphor bronze was supplemented by additional data.

Acknowledgments:

The authors acknowledge the interest of D. K. Crampton, Director of Research of the Chase Brass and Copper Co., in this creep testing program and the assistance of those who participated in various phases of the work. Permission to publish the results was given by the Chase Brass and Copper Co.

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DISCUSSION

Mr. P. G. McVetty.[1]—I have one question concerning the interpretation of curves showing the relation between stress and creep rate. Several alloys including arsenical copper, admiralty and aluminum brass, copper-nickel-phosphorus and copper-nickel-phosphorus-tellurium alloys give curves which turn down at the left-hand end in the double-logarithmic plot. When this method of plotting was introduced by F. H. Norton many years ago,[2] he expressed some doubt about continuing a straight line to lower rates of creep. It is these lower creep rates and lower stresses in which the designer is usually interested when data of this type are applied to the use of alloys at elevated temperatures. I should like to ask whether the authors have tried the semi-logarithmic plot and the hyperbolic sine relation suggested by Nadai. It has been shown[3] that this relation has both theoretical and experimental support and it leads to more conservative stress estimates than the straight-line extrapolation to lower stresses in the double-logarithmic plot. The application of this method to any constant-temperature stress-creep rate data is facilitated by the use of charts.[4]

Mr. H. L. Burghoff (*author's closure*).—We have not plotted the stress-creep rate relationships in the semi-logarithmic manner which Mr. McVetty has indicated but have confined ourselves to the fully logarithmic plots. With regard to departure from linearity of some of these curves, as evidenced by their turning down in the region of low stress and low creep rate, the hyperbolic sine relation may possibly provide a satisfactory interpretation of the results. However, there remains the possibility that the curvature in question is at least partly associated with the character of the primary creep curves. As has been mentioned, most of the creep curves represented in the present paper still showed decreasing rates of creep at the end of the tests. In a previous analysis of our creep data on a phosphorized copper (reference 2), it was found that the logarithmic plots of stress and creep rate became more nearly straight as the primary creep curves more nearly approached the condition of constant creep rate.

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[1] Manager, Strength of Materials Section, Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

[2] F. H. Norton, "The Creep of Steel at High Temperatures," McGraw-Hill Book Co., Inc., p. 16 (1929).

[3] P. G. McVetty, "Creep of Metals at Elevated Temperatures—The Hyperbolic Sine Relation Between Stress and Creep Rate," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 65, pp. 761–769 (1943).

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WELDING OR STICKING OF ELECTRICAL CONTACTS

By Erle I. Shobert, II[1]

Subcommittee X of A.S.T.M. Committee B-4 on Electrical Heating, Resistance, and Related Alloys has been working for about four years on a machine to test the properties of contact materials under conditions independent of a particular piece of commercial apparatus. There have been many problems to be solved in working out a standard test machine, and the group under the chairmanship of B. W. Jones has done a remarkable job.

One problem which has received considerable attention has been a method of measuring the welding or sticking characteristics of contacts on the machine. As will become apparent in the discussion which follows, a measurement of this type is particularly difficult because the mechanical factors such as impact, bounce, and vibration play a big part in the development of sticking and these are particularly difficult to control in the design and manufacture of the individual machines.

Working on this angle of the problem, the group finally found a method of setting up the various test machines in such a manner that consistent results on welding and sticking were secured from six different machines operating in the laboratories of Baker & Co., Callite Tungsten Corp., The General Electric Co., The General Plate Division of Metals and Controls Corp., P. R. Mallory & Co., and the Westinghouse Electric Corp.

This work has been carried out by the following members of the main committee: B. W. Jones, *Chairman*, General Electric Co.; F. E. Carter, Baker & Co.; George Durst, General Plate Div.; S. G. Eskin, Robertshaw Thermostat Co.; A. B. Gibson, Gibson Electric Co.; F. R. Hensel, P. R. Mallory & Co.; J. D. Kleis, Fansteel Metallurgical Corp.; H. M. Lusk, Callite Tungsten Corp.; Wm. Pakala, Westinghouse Electric Corp.

At the November, 1945, meeting of the committee, Mr. Jones presented the data shown in Fig. 1 to the committee as the result of the effort to standardize the sticking and welding tests on the machines. It can readily be seen that the agreement among the tests on different machines in the various laboratories is remarkable. This is the first time that any such consistent data on welding or sticking of electrical contacts have been available.

It appeared that there must be some fundamental physical basis for the different values of the limiting current as shown in Fig. 1, and perhaps for the first time, we have the experimental data required to verify a theory describing the phenomenon of contact welding. The discussion which follows is an attempt to find this theory and to verify it by means of the tests described in Fig. 1 and by other subsequent tests.

Presentation of Data

The test machine is described in detail in A.S.T.M. Tentative Method for

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[1] Vice-Chairman, A.S.T.M. Committee B-4 on Electrical Heating, Resistance, and Related Alloys; Stackpole Carbon Co., St. Marys, Pa.

Life Test of Electrical Contact Materials (B 182 – 46 T)[2] and in a paper by A. M. Suggs (**1**).[3] A revision of this method is now in progress and will be published soon. The test procedure will not be described in detail; however, it will be discussed to the extent required for the proper interpretation of the test results.

Three sets of contacts on the machine are started out at a current which is expected to give a certain number of welds or sticks. If the desired result is not achieved, the current is increased or decreased and successive tests are run until the point at which welding starts is determined. The no-weld or limiting current is the highest current at which the contacts will operate 100,000 times without welding.

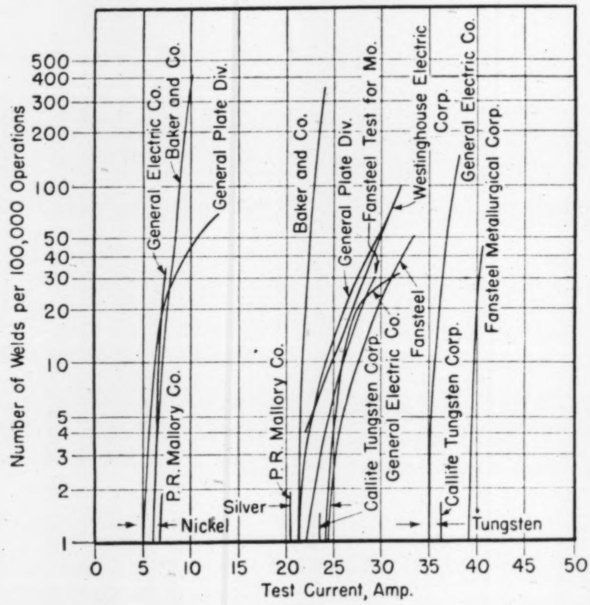

FIG. 1.—Welding Characteristics of Electrical Contact Materials as Determined by the A.S.T.M. Contact Testing Machine.

The machine is designed to shut itself down when one set of contacts sticks or welds, so that the operator not only knows about each weld, but may also measure the strength of the weld by pulling the contacts apart with a spring balance.

The following test conditions were used to get the data shown in Fig. 1, and for all the tests on limiting current described in this paper: voltage E_1—115 v., 60-cycle a. c.; limiting current I_1—as described; load—resistance load, unity power factor; closing force F—100 g. or 0.22 lb.; opening force F_1—75 g. or 0.165 lb.; opening and closing contact velocity v_0—1.5 in. per second (3.82 cm. per second); stationary contact $\frac{1}{4}$-in. diameter — flat — high initial polish; moving contact $\frac{1}{4}$-in. diameter with $\frac{5}{8}$-in. radius, b, on face—high initial polish; operating speed 60 operations per minute.

All tests on silver and nickel were run on special contacts prepared by P. R.

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[2] 1946 Book of A.S.T.M. Standards, Part I-B, p. 711.

[3] The boldface numbers in parentheses refer to the references appended to this paper, see p. 767. A comprehensive Bibliography on Electrical Contacts has been published by the A.S.T.M. The Bibliography and the yearly supplements thereto may be secured from the A.S.T.M. Headquarters.

Mallory Co. for this purpose. Other tests were made on contacts prepared by the different investigators.

The limiting currents or no-weld currents taken from Fig. 1, reported by Mr. Jones, are given in Table I. These values and the data on the test conditions will be used in the presentation which follows.

THEORY

There is practically no published information on the welding or sticking of electrical contacts which applies to the problem at hand. It is therefore necessary to develop a theory to fit the experimental facts with little reference to previous work.

The first point to be noticed from Fig. 1 is that when welding starts, as the current is increased, it increases very rapidly. This means that the limiting current as determined by experiment is a fairly definite quantity. The agreement among values of the limiting current measured by the different investigators indicates, as pointed out above, that there must be some reason, based upon the physical properties of the materials, for the effects observed.

TABLE I.—LIMITING CURRENTS.

Contact Material	Limiting Current I_1 amp.
Silver	21
Nickel	5
Tungsten	35

As to mechanism of the welding effect, it is necessary to make certain reasonable assumptions. These are as follows:

1. It is assumed that welding or sticking, when it occurs, takes place either on opening or closing the circuit due to the heat generated in the contact material during the time the contact resistance is building up or decreasing.

2. It is assumed that near the limiting current, this welding takes place only when the contacts happen to open or close near the current and voltage peak (see Appendix).

3. It is assumed that when a weld occurs, it is confined to one spot on the contact surface and not to several regions. This assumption is based upon some of the reasoning given in an earlier paper by the author (2). Formula 9 in reference 2 shows that the contact resistance is highest when the number of contact areas, n, is 1. This also means that when n = 1 there will be more energy dissipated in the contacts under a given set of circuit conditions and that this energy will all be dissipated in one continuous region on the contact surface. This partly explains why the contacts do not weld on every operation after the limiting current is reached.

4. It is assumed that near the limiting current, a weld occurs only when two points of clean metal from each surface come in contact. The presence of oxide films or any nonwelding metallic surface impurities will tend to eliminate the chance of welding under a particular set of circumstances.

5. It is assumed that the contacts may actually stick or weld many more times than are indicated by the stopping of the machine, but that only those welds are indicated which are strong enough to hold together under the application of the opening force. This means that a solid bridge of metal is formed whose cross-section is greater than that determined by the opening force and the tensile strength, S, of the material.

6. It is assumed that the points of metal which come in contact to form the welds are completely annealed and are in the softest form possible. This assumption is reasonable because temperatures at or near the melting point of the contact material are possible and

continued operation of the contacts must heat up the small areas of contact to these temperatures.

If assumptions 2, 3, 4, and 5 are correct, it can readily be seen that when sticking starts, it is of the order of from 1 to 10 welds per 100,000 operations. It is not possible from the data available at present to calculate the probabilities in this case, but it is clearly evident that every operation cannot produce a weld or stick just as soon as the limiting current, as here defined, has been exceeded. This point is discussed more in detail in the appendix.

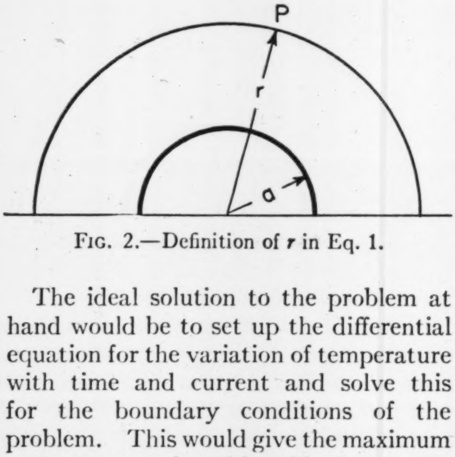

FIG. 2.—Definition of r in Eq. 1.

The ideal solution to the problem at hand would be to set up the differential equation for the variation of temperature with time and current and solve this for the boundary conditions of the problem. This would give the maximum temperature to be achieved in the contact surface under the transient conditions during contact opening and closing similar to the steady state solution described in reference 2, Eq. 17. Holm (3) has given the differential equation for the variation of temperature with time and position in the contacts when the contacts are closed and the current turned on by another switch. This equation is:

$$\frac{d^2T}{dr^2} + \frac{2dT}{rdr} + \frac{1}{r^4}\frac{\rho i^2}{4\pi^2\lambda} = \frac{Q}{\lambda}\frac{dT}{dt} \quad \ldots (1)$$

where r is defined in Fig. 2, T is the temperature at the point P, ρ is the specific resistance of the material, λ is the heat conductivity of the material, Q is the specific heat, and i is the current. When the variation of i with time is included in this equation, it becomes impossible to get any simple solution. In addition, the results would only be approximate unless the variations of ρ, λ, and Q with temperature were also considered.

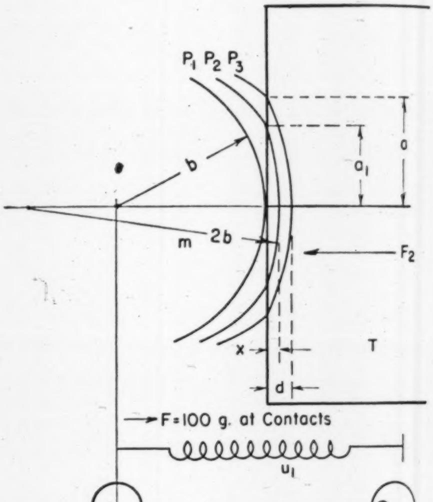

FIG. 3.—Calculation of t_1.

Another approach to the problem is to find the energy dissipated in the contacts by the electrical circuit during the opening or closing time and compare this with the amount of energy required to melt a bridge of material sufficient to make a weld.

By assuming that the resistance in the circuit varies as the function

$$\frac{R_1 t_1}{t_1 - t}, \text{ where } t_1 \geq t \geq 0,$$

and where R_1 is the resistance in series with the contacts and t_1 is the total time required to complete the make or break, J. A. Wilcken (4) shows that the energy dissipated in the contacts during the opening or closing cycle on a noninductive circuit without arcing is

$$W_1 = \frac{E_1^2}{6R_1} t_1 \ldots\ldots\ldots\ldots (2)$$

where W_1 is the energy in watt-seconds and E_1 is the open circuit voltage.

Our problem now is to find a value for t_1 for the different contact materials under consideration. A. M. Suggs (1) shows the construction of the A.S.T.M. machine and the way the contacts operate. The details of the operation are also shown in Fig. 3, where a contact of effective mass m and contact radius b is shown at three points of penetration P_1, P_2, and P_3 into the stationary contact T. The contact spring U_1 provides a force of 100 g. at the contacts. The cam C through the rod L provides for the motion of the moving contact. The spring U_2 with tension $F + F_1$ provides the opening force F_1 when the rod L is riding up the cam in opposition to spring U_1. The velocity of the moving contact is v_0. a_1 is the radius of the contact area at the penetration x, and a is the radius of the contact area at the penetration d. The penetration d is determined by the contact yield point, Y, of the material, the contact force, and the radius of the contact surface as follows:

Since (2)

$$F = \pi a^2 Y \ldots\ldots\ldots\ldots (3)$$

and

$$d = \frac{a^2}{4b} \ldots\ldots\ldots\ldots (4)$$

we have

$$d = \frac{F}{4\pi b Y} \ldots\ldots\ldots\ldots (5)$$

where we have assumed that half of the deformation has taken place on each contact, doubling the radius of the moving contact.

The motion of the moving contact arm is controlled by the contact springs U_1 and U_2 and the cam C. We will consider all the forces as though they were operating at the contacts and consider that they have been corrected to the contacts for the position along the contact arm and the distance from the fulcrum. The spring U_1 develops a force F equal to 100 g. which is constant over the distances considered in this discussion. Before the contacts touch, this force is taken up by the rod L and the cam C. After the contacts come together, moving with the velocity v_0, part of the force F is taken up by the contacts, so that

$$F = F_2 + F_3 \ldots\ldots\ldots\ldots (6)$$

But $F_2 = \frac{\pi a_1^2}{\pi a^2} F$, since the area πa^2 supports the force F. Or also

$$F_2 = \frac{x}{d} F \ldots\ldots\ldots\ldots (7)$$

thus at $x = d$, $F_3 = 0$ and the full force F is applied to the contacts.

During this time, however, the contacts have been moving at the velocity v_0. It is now necessary to consider the over-travel due to the inertia of the mass of the contact arm. The kinetic energy of the contact arm is $\frac{mv_0^2}{2}$ and the energy dissipated by the moving contact in penetrating further into the stationary contact is

$$\int_d^x F_2 \, dx$$

when plastic deformation of the material is assumed. This assumption is shown to be reasonable in reference (2).

Equating these values of the energy, we have

$$\tfrac{1}{2} m v_0^2 = \int_d^x F_2 \, dx = \frac{F}{d} \int_d^x x \, dx \ldots (8)$$

or

$$\tfrac{1}{2} m v_0^2 = \frac{1}{2} \frac{F}{d} (x^2 - d^2) \ldots\ldots\ldots (9)$$

This becomes

$$x = \sqrt{\frac{mv_0^2\, d}{F} + d^2} \quad \text{.........(10)}$$

Under the conditions of the A.S.T.M. test machine, $m = 100$ g., $v_0 = 1.5$ in. per second, and $F = 100 \times g$ ($g = 386$ in. per second per second). Thus

$$x = \sqrt{\frac{(1.5)^2\, d}{386.} + d^2} \quad \text{.......(11)}$$

For silver, $d = 0.0006$ in. so that $x = 0.0020$ in. The over-travel is thus 0.0014 in.

Wilcken's work (4) does not consider this over-travel or its effect upon the time required to make or break a contact circuit. His formula applies only to that interval of time between initial contact and when full contact is reached. It is readily seen, however, that practically all of the energy dissipated in the contacts is dissipated in the period of time between the initial contact and when the contacts reach the penetration d. The same holds true when the contacts are opening. Thus, t_1 may be determined simply by the relation

$$t_1 = \frac{d}{V_0} \quad \text{.............(12)}$$

This gives a figure which is applicable to Wilcken's work and which is reasonably close to the actual case for our immediate purpose. Combining Eqs. 2 and 12, we have

$$W_1 = \frac{E_1^2}{6R_1} \cdot \frac{d}{V_0} \quad \text{.........(13)}$$

But $d = \frac{a^2}{4b} = \frac{F}{4\pi b Y}$,

since $F = \pi a^2 Y$, so that

$$W_1 = \frac{E_1^2}{6R_1} \cdot \frac{F}{4\pi b v_0 Y} \quad \text{.........(14)}$$

This may also be written

$$W_1 = \frac{E_1 I_1 F}{24\pi b v_0 Y} \quad \text{..........(15)}$$

The A.S.T.M. tests described above were run at 115 v. 60-cycle a.c. Since it is assumed that welding occurs when the contacts close on the voltage peak (also current peak with pure resistance load) the value of E_1 to be placed in Eq. 15 is $1.41 \times 115 = 162$ v. I_1, of course, is the limiting current in amperes and its peak value must be used here. Under the conditions described, the energy is that generated as the contacts close.

Placing the values of E_1 (162-v. peak), I_1 (limiting current, peak, calculated from Fig. 1), F (0.22 lb.), b (0.625 in.), v_0 (1.5 in. per second), and Y (contact yield point) in Eq. 15, we find the values for the energy W_1 listed in Table II.

TABLE II.—ELECTRICAL ENERGY DISSIPATED IN CONTACTS DUE TO CHANGE IN CONTACT RESISTANCE ON CLOSING.

Material	W_1 (calculated from Eq 15), watt-sec.
Silver	0.33×10^{-3}
Nickel	0.020
Tungsten	0.037

The contact yield point is listed in Table VIII where the value for the annealed or soft material is used. These values are also found in reference 2 Table I.

We will now estimate the energy W_2 required to heat a sphere of the contact material of radius a to the melting point. This is the same as assuming that the whole contact area πa^2 is brought to the melting point. We will also assume that $n = 1$, or that there is only one area of contact. We find then that

$$W_2 = (2.54)^3 \times \frac{4\pi}{3} a^3 \times Q_{av} \times T_m \text{ watt-sec.} \quad \text{.(16)}$$

Where Q_{av} is the estimated average specific heat in watt-seconds per cubic centimeter over the range from room temperature to the melting point of the material T_m, and where the proper factors have been added to give the result in the described units. Table III shows the value of W_2 calculated for the three materials. The radius, a, of the contact area is calculated from Eq. 1, reference 2.

TABLE III.—ENERGY REQUIRED TO BRING A SPHERE OF RADIUS EQUAL TO THE RADIUS OF THE CONTACT AREA TO ITS MELTING POINT.

Material	a, in.	Q_{av}, Watt per sec. per cu. cm.	T_m, deg. Cent.	W_2, watt per second
Silver.......	1.23×10^{-3}	2.62	961	0.322×10^{-3}
Nickel......	0.633	4.85	1452	0.119
Tungsten....	0.324	2.74	3300	0.0212

When the calculated values of W_1 and W_2 are compared for the contact materials listed, it is seen that while the results are of the correct order of magnitude, there is no correlation in the way the quantities vary from one material to another. This indicates that the problem is more complicated than the mechanism proposed describes, but that some similar effect or effects must be taking place.

TABLE IV.—ENERGY REQUIRED TO BRING A SPHERE OF RADIUS a_2 TO THE MELTING POINT.

Material	S, psi.	a_2, in.	W_3, watt-sec.
Silver..........	42 000	1.12×10^{-3}	0.24×10^{-3}
Nickel..........	155 000	0.583	0.096
Tungsten.......	590 000	0.299	0.017

It is also possible to estimate the energy required to melt down a bridge of material whose area would be that required to break under the opening force based on the tensile strength of the material. The results of these calculations are given in Table IV, where the energy required to melt a sphere of material of radius a_2 is calculated for the contact materials under consideration. In this case a_2 is the radius of the cross-section which is calculated from the opening force $F_1 = 75$ g. and the tensile strength S of the material, where

$$F_1 = \pi a_2^2 S \qquad (17)$$

Thus this energy, W_3, is

$$W_3 = (2.54)^3 \times \frac{4\pi}{3} a_2^3 \times Q_{av} \times T_m \text{ watt-sec.} \qquad (18)$$

Comparing W_2 in Table III and W_3 in Table IV, it is seen that W_3 is between 75 and 80 per cent of W_2. This is because there is a fairly uniform relation between the tensile strength and the contact yield point. The correlation tween W_3 in Table IV and W_1 in Table II is no better than between W_2 and W_1.

TABLE V.

Material	H_f, watt-sec. per cu. cm.	W_4, watt-sec.
Silver	1070	0.0136×10^{-3}
Nickel	2760	0.0048
Tungsten..................	....	....

The heat of fusion might be expected to play some part in the process of welding or sticking of contacts. If it is assumed that 10 per cent of the volume of a sphere of radius a is melted, this energy W_4 may be calculated from Eq. 19.

$$W_4 = 0.1 \times (2.54)^3 \times \frac{4\pi}{3} a^3 \times H_f \text{ watt-sec.} \qquad (19)$$

where H_f is the heat of fusion at the melting point in watt-seconds per cubic centimeter. Table V shows the values of W_4 and H_f.

A comparison of the data in Table V with that in Table II shows that the heat of fusion is not an important factor. The choice of 10 per cent of the volume of the sphere is somewhat arbitrary, but to maintain the full volume of the sphere of radius a at the melting point would require an extended period of time at low contact force and a very high contact drop. The requirements for this will be discussed below.

To this point we have not considered the specific resistance of the material ρ, the heat conductivity λ, nor their variation with temperature. In reference 2, Eqs. 11 to 17, is derived a method of estimating the maximum contact temperatures when the temperature rise is low and when the variation in ρ and λ with temperature are neglected.

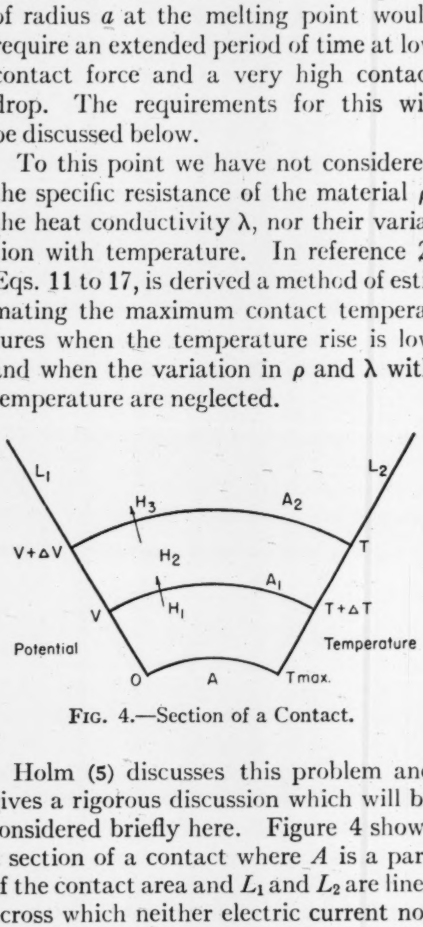

FIG. 4.—Section of a Contact.

Holm (5) discusses this problem and gives a rigorous discussion which will be considered briefly here. Figure 4 shows a section of a contact where A is a part of the contact area and L_1 and L_2 are lines across which neither electric current nor heat flows. Holm shows that if it is assumed that the flows of heat energy and electric current are parallel the temperature and potential are uniquely determined at every point. Thus, the surfaces, A, A_1, and A_2 are surfaces over which the voltage and temperature are constant for each surface. This condition is reasonably satisfied in most contact applications, since these effects take place in a thin section of the contact material near the contact surface (2). It is also assumed that T_{max} is the highest temperature in the contact surface and that this represents a true maximum, that is, $\frac{dT}{dr} = 0$. Pure metallic contact is also assumed, since if a high-resistance film were present, $\frac{dT}{dr}$ could not be zero at the contact surface.

Consider, now, two equipotential surfaces A_1 and A_2 of potential V and $V + \Delta V$ and temperature $T + \Delta T$ and T. Let the electrical resistance between these surfaces be ΔR and the thermal resistance ΔZ. Let us also choose the surfaces so that ΔR is the same between all of the surfaces chosen.

Let H_1 be the heat flowing across A_1, H_2 the heat generated by the current flow between A_1 and A_2, and H_3 the heat flowing out across A_2. Then

$$H_1 + H_2 = H_3 \quad \ldots\ldots\ldots (20)$$

Or

$$-\frac{\Delta T}{\Delta Z} + \frac{(\Delta V)^2}{\Delta R} = -\frac{\Delta(T + \Delta T)}{\Delta(Z + \Delta Z)}$$

$$= -\frac{\Delta T + \Delta^2 T}{\Delta Z + \Delta^2 Z} \quad \ldots (21)$$

but

$$\Delta^2 Z \ll \Delta Z,$$

therefore,

$$\frac{1}{\Delta Z + \Delta^2 Z} = \frac{1 - \frac{\Delta^2 Z}{\Delta Z}}{\Delta Z}$$

so that,

$$-\frac{\Delta^2 T - \Delta T \frac{\Delta^2 Z}{\Delta Z}}{\Delta Z} = \frac{(\Delta V)^2}{\Delta R} \quad \ldots (22)$$

But

$$\frac{\Delta Z}{\Delta R} = \frac{1}{\rho\lambda} \quad \ldots\ldots\ldots\ldots (23)$$

and ΔR is a constant, so that

$$\Delta^2 Z = \Delta R \cdot \Delta\left(\frac{1}{\rho\lambda}\right) = \frac{\Delta R}{(\rho\lambda)^2}\,\Delta(\rho\lambda) \,..\, (24)$$

Placing this in Eq. 22 above, we have

$$\frac{(\rho\lambda)\Delta^2 T \;+\; \Delta T \Delta(\rho\lambda)}{\Delta V} = \Delta\left(\rho\lambda\,\frac{\Delta T}{\Delta V}\right)$$

$$= -\,\Delta V \,..\, (25)$$

Taking the limits in Eq. 25, we have,

$$d\left(\rho\lambda\,\frac{dT}{dV}\right) = -dV \,\ldots\ldots\ldots\, (26)$$

Integrating this and applying the limits for the A surface, where we have $T_{\max}$, $\frac{dT}{dV} = 0$, and $V = 0$, we have

$$\rho\lambda\,\frac{dT}{dV} = -V \,\ldots\ldots\ldots\ldots\, (27)$$

The second integration gives

$$\int_{T'}^{T_{\max}} \rho\lambda\, dT = \tfrac{1}{2}V^2 \,\ldots\ldots\ldots\, (28)$$

But $V = \frac{E}{2}$, where E is the total voltage across two contacts, so we may write

$$\int_{T_c}^{T_{\max}} \rho\lambda\, dT = \frac{E^2}{8} \,\ldots\ldots\ldots\, (29)$$

where T_c is measured back at the point where E is measured, and both are close enough to the contact surface so that the original conditions of the derivation are fulfilled, that is, that the current and heat flow are colinear.

As Holm (5) shows, if we apply the Franz Wiedeman law $\rho\lambda = K\underline{T}$ where $\underline{T}$ is in degrees Kelvin, we have

$$\tfrac{1}{2}K(\underline{T}_{\max} - \underline{T}_c^2) = \frac{E^2}{8} \,\ldots\ldots\ldots\, (30)$$

This may also be written

$$I_1^2 R_2^2 = 4K(\underline{T}_{\max}^2 - \underline{T}_c^2) \,\ldots\ldots\, (31)$$

where I_1 is the limiting current and R_2 is the contact resistance. Since I_1 as measured is the root mean square value of the current, it will be considered as such from now on in the discussion. To use the peak value would only change the value of the constants and might confuse the issue. If we substitute the melting point of the material $\underline{T}_m$ for $\underline{T}_{\max}$, and room temperature for T_c, we have

$$R_2^2 = \frac{4K(\underline{T}_{\max}^2 - \underline{T}_c^2)}{I_1^2} \,\ldots\ldots\ldots\, (32)$$

Values of R_2 for the three materials under consideration are given in Table VI.

TABLE VI.

Material	R_2 (Eq. 32), ohms	R (reference 2, Eq. 9), ohms
Silver	1.72×10^{-2}	2.61×10^{-4}
Nickel	8.44	24.5
Tungsten	3.45	33.9

Also given in Table VI are values of the contact resistance R calculated from reference 2, Eq. 9, using the conditions described for the A.S.T.M. contact test machine. This is the contact resistance when the contacts are closed under the full force. The values of resistance are of a different order of magnitude, as would be expected, and they also show no correlation in the way they vary. The resistance R_2 is in effect the contact resistance required to melt the contact points when the limiting current is flowing under steady state conditions. Such a mechanism is still far from the conditions which exist when contacts open or close on an electric circuit.

Up to this point, we have considered several aspects of the problem, and while the agreement in order of magnitude is reasonable, the factors considered do not appear to account for the relative differences between the materials. It thus appears that an empirical approach to the problem might throw some light on the situation if such an empirical relation can be worked out. The approach to the problem which follows is the result of a series of investigations which in themselves were unsuccessful, but which lead to the method to be described.

If we start with Eq. 31 and consider some of the other variables which influence the results, we may write a general equation as follows:

$$I_1^2 \propto KT_m S^{-1} \rho_{av}^{\alpha} Y^{\beta} Q_{av}^{\gamma} \quad \text{.......(33)}$$

TABLE VII.—COMPARISON BETWEEN CALCULATED AND MEASURED VALUES OF THE LIMITING CURRENT I_1.

Material	I_1, measured, amp.	I_1, calculated from Eq. 37, amp.
Silver	21	20.8
Nickel	5	4.9
Tungsten	35	37
Palladium	5[a]	5.3
ES110 (silver alloy)	12[b]	13.6
Platinum	15[g]	8.8
Molybdenum	20[f] 22[h]	21.8
85Ag-15Cd	20	...[c]
75Ag-25Zn	4.4	...[c]
Graphite	...[d]	...[e]

[a] This value was measured before the machine was in its final adjustment. The value may be somewhat low. Reported by B. W. Jones, General Electric Co.

[b] This alloy contains 85 per cent silver. Values for silver of the temperature coefficient of resistivity and O_{av} were assumed. All other properties were measured. Reported by B. W. Jones, General Electric Co.

[c] There are not sufficient data to calculate these values.

[d] It has not been possible to weld graphite under any of the conditions available on practical equipment.

[e] The absence of a melting point under atmospheric conditions makes it impossible to calculate the limiting current. Since no liquid phase is present, no welding can occur.

[f] Reported by B. W. Jones, General Electric Co.

[g] Reported by K. C. Underhill, Baker & Co. Platinum appears to be the only material measured to date which does not fit into the calculations. The reason is not apparent.

[h] Reported by J. D. Kleis, Fansteel Metallurgical Corp.

where ρ_{av} is the average value of the specific resistance between room temperature and the melting point, Q_{av} is the average value of the specific heat in watt-seconds per cubic centimeter between room temperature and the melting point, Y is contact yield point (annealed), and α, β and γ are constants to be determined. The exponent of S is chosen as -1 based on the data given in Table IV. We may also write Eq. 33 as follows:

$$I_1^2 = GK\underline{T}_m^2 S^{-1} \rho_{av}^{\alpha} Y^{\beta} Q_{av}^{\gamma} \quad \text{......(34)}$$

where G is a constant for the A.S.T.M. test machine under the conditions described for these tests. Our problem now is to find values for α, β, and γ which will satisfy this equation for silver, nickel, and tungsten, and then to check the equation on other materials for which test data are available. Trial and error shows that $\alpha = -2, \beta = 2.5$ and $\gamma = -2$ are reasonable values.

Placing these values of α, β and γ in Eq. 34, we have

$$I_1^2 = \frac{GK\underline{T}_m^2 Y^{2.5}}{\rho_{av}^2 Q_{av}^2 S} \quad \text{.........(35)}$$

and

$$G = \frac{I_1^2 \rho_{av}^2 Q_{av}^2 S}{K\underline{T}_m^2 Y^{2.5}} \quad \text{..........(36)}$$

Substituting the proper values of the quantities in Eq. 36 we have

Silver $G = 2.48 \times 10^{-14}$
Nickel $G = 2.63 \times 10^{-14}$
Tungsten $G = 2.14 \times 10^{-14}$
Average $G = 2.42 \times 10^{-14}$

Using this average value for G, Eq. 35 now becomes

$$I_1^2 = \frac{2.42 \times 10^{-14} K\underline{T}_m^2 Y^{2.5}}{\rho_{av}^2 Q_{av}^2 S} \quad \text{.....(37)}$$

The calculated and experimental results are compared in Table VII. Some additional tests which were run on the A.S.T.M. machine by various members of the committee are also reported. Where sufficient data were available, the calculated values of I_1 are also given. The physical properties used in the calculations are all given in Table VIII which is a repetition in part of the information given in Table I of reference 2.

As is to be expected, the agreement between measured and calculated values of I_1 for Ag, Ni, and W is very good. The verification of the theory based on the tests on palladium, molybdenum, and ES110 is within the limits of error which might be expected in the tests and in the values of the physical properties. Platinum is an exception in which the measured value of the limiting current is almost twice the calculated value. The reason for this difference is not apparent.

Graphite does not fit into the calculations because it is known to sublime under atmospheric pressures (6). Thus there is no melting point or molten phase present to cause welding or sticking. While it does not check or confirm the calculations, it tends to bolster the theory that the melting point is important in the phenomenon of welding or sticking on electrical contacts under the conditions described.

TABLE VIII.—PHYSICAL PROPERTIES OF CONTACT MATERIALS.
All values not otherwise noted taken from standard tables.

Material	Specific Gravity	Melting Point deg. Kelvin	Specific Resistance, ρ, ohm-in.	ρ_{av}, ohm-in.	Specific Heat Q_{av}, watt-sec. per cu. cm.	*Y* Contact Yield Point, psi.		*S* Tensile Strength, psi.	λ Heat Conductivity, watts per inch per deg. Cent.	Constant *K* in Franz Wiedemann Law $\rho\lambda = KT$
						Annealed	Hard			
Silver	10.4	1234	0.64×10^{-6}	1.78×10^{-6}	2.62	46 000	68 000	42 000	10.64	2.27×10^{-8}
Nickel	8.9	1725	3.1	13.0	4.85	175 000	......	155 000	1.49	1.54
Tungsten	19.3	3573	2.2	23.4	2.74	665 000	......	590 000	4.07	2.98
Palladium	11.4	1728	3.94^c	15.8	3.24	90 000^c	250 000^c	54 000	1.71^c	2.25^c
ES110 (Silver alloy)	9.55^b	1123^b	1.37^b	3.7	2.40	66 000^b	160 000^b	55 000^b	......	2.27^a
Platinum	21.5	2028	3.87^c	15.9	3.33	105 000^c	230 000^c	35 000	1.77^c	2.28^c
Molybdenum	10.2	2893	2.2	16.6	3.20	350 000^e	430 000^f	260 000	3.71	2.72
Graphite	2.2	....^{d}	320.	440.^{b}	4.6	......	6 700^b	2 500^b	0.399	36.2

[a] Assumed to be the same as that of silver.
[b] Measured in laboratory of the Stackpole Carbon Co.
[c] Supplied by F. E. Carter, Baker & Co.
[d] Graphite sublimes at temperatures near 4000 K. under ordinary atmospheric pressure (6).
[e] From P. R. Mallory Catalog. Hardness Rockwell B90 or Rockwell 45T62. This is the value for molybdenum rod.
[f] This value is shown in Reference 2, Table I and was measured on drawn wire.

The application of Eq. 37 to d-c. circuits would require a value of *G* twice that for a.c. since it is the peak current which causes welding.

DISCUSSION

There are several questions which should be raised concerning the application of the theory. In the first place it must be checked on some of the other materials for which satisfactory data are available on the physical properties. More information on contact alloys and sintered materials would be of interest. If these also fall in line, the theory can be considered acceptable.

The question might be raised as to why the specific heat, Q_{av}, appears as it does in an inverse relation to the limiting current. Casual observation would indicate that the higher the specific heat, the higher the limiting current should be, since more energy would be required to bring the contact area to the melting point. This method of looking at the problem, however, does not bring time into consideration. It is possible that a low specific heat means that in the instant of contact, more of the material will be softened, the contact area will increase more rapidly to the point where melting temperatures are no longer possible, and therefore less total energy will be dissipated in the contacts without the formation of a weld. An investigation of the oscillograms of the contact make and break might throw additional light on this point.

The specific heat may also enter into the problem from the standpoint of the condition of the contact surfaces after the contacts have operated for a considerable number of times. A lower specific heat might result in smoother, more rounded projections on the contact surface due to localized melting and thus result in a higher value of the limiting current. The question of the specific heat is particularly difficult to check because the specific heat cannot be changed without changing other important characteristics of the contacts.

It became apparent to the author, when looking for data to use to verify the theory, that there is room for a considerable amount of work to be done on the physical properties of the materials used for contacts. Almost all of the factors discussed in the paper are open to question in some way or other. To get the complete story on a problem like this, the following information should be worked out and methods determined for finding these data at all temperatures from room temperature to the melting point.

1. Resistivity.
2. Heat conductivity.
3. Specific heat.
4. Contact yield point or hardness.
5. Tensile strength.

This information and methods of measuring it should have application in other fields and would not be limited to contacts alone. If better data were available, it might be that better results would be achieved using values of the resistivity and heat capacity at the melting point, or at some point near the melting point. The same applies to the mechanical properties.

The constant G in Eq. 35 is worked out for a particular set of test conditions on the A.S.T.M. machine. If the opening and closing forces and opening and closing speeds are changed, the value of G will change. It is to be expected that G would increase with increased opening and closing forces and with increased opening and closing speeds, although the method of variation may not be simple. A few tests run at different forces and speeds would show how this works out and would give valuable information to designers of electrical equipment who must consider the problems of sticking and welding. Tests should also be run to see if the value of G for d.c. is twice that for a.c. as would be expected.

SUMMARY

1. A formula has been developed to show the relationship between the limiting current on the A.S.T.M. testing machine and the physical properties of contact materials. If this receives further verification, it should lead to a practical solution of many of the welding and sticking problems on contacts.

2. It would be desirable to have data and develop test methods to show the resistivity, heat capacity, heat conductivity, and mechanical properties of contact metals and materials from room temperature to their melting points.

3. Further tests should be made on the machine to check the value of the constant G in Eq. 35 and see how it varies with opening and closing speeds and opening and closing forces.

4. The nonwelding properties of graphite are probably due to the fact that it has no melting point under atmospheric conditions.

5. A reasonable explanation of the steepness of the curves in Fig. 1 is given which shows that the contacts weld only once in every 200 to 2000 times when a weld might be possible.

Acknowledgments:

The consistent data obtained upon four different machines in four different laboratories made possible the development of a reasonable theory to describe the welding phenomenon. In fact, with-

out this experimental work, the development of a theory of any kind would be impossible. Thus due credit should be given the group who has made this or any theoretical work possible.

Additional tests on the contact test machine and data on some of the contact materials were supplied by B. W. Jones of the General Electric Co., K. C. Underhill of Baker and Co., and J. D. Kleis of the Fansteel Metallurgical Corp. These tests and data made possible the partial confirmation of the theory with palladium, molybdenum, and the silver alloy ES110.

F. E. Carter of Baker and Co. supplied most of the data on the physical properties of the platinum metals.

The Engineering Laboratory of the Stackpole Carbon Co. investigated and measured the properties of several of the materials.

Appreciation is expressed to the management of the Stackpole Carbon Co. for permission to publish this work.

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All of the tests which have been reported on the A.S.T.M. machine have been run on a.c. The question arises as to the relation between the current or voltage cycle and the slopes of the curves of limiting current shown in Fig. 1.

It has been assumed that when welding occurs it takes place near the current and voltage peak. We also know from the characteristics of the machine, the driving motor, cams, etc., that the operation is purely random as to the point on the a-c. cycle at which opening and closing takes place.

By comparing the length of time during a cycle that the current exceeds the limiting current with the length of the cycle, we have an estimate of the comparative number of operations when a weld might be possible. Comparing this data with the test results will show us how many welds actually occur out of these possibilities.

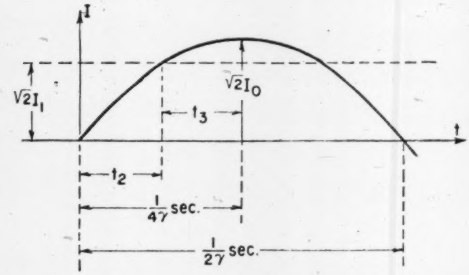

FIG. 5.—One Half Cycle of a-c. Current.

Figure 5 shows a single half cycle of a-c current of frequency ν (in this case 60 cycles). I_1 is the limiting current whose peak value is $\sqrt{2}\,I_1$. The current I has the peak value of $\sqrt{2}\,I_0$. We have

$$I = \sqrt{2}\,I_0 \sin 2\pi\nu t$$

However, at t_2, $I = \sqrt{2}\,I_1$, so that

$$\sqrt{2}\,I_1 = \sqrt{2}\,I_0 \sin 2\pi\nu t_2$$

Thus

$$t_2 = \frac{1}{2\pi\nu} \sin^{-1} \frac{I_1}{I_0}$$

and

$$t_3 = \frac{1}{4\nu} - \frac{1}{2\pi\nu} \sin^{-1} \frac{I_1}{I_0}$$

Let us call J the ratio of the time the current exceeds $\sqrt{2}\,I_1$ to the total time.

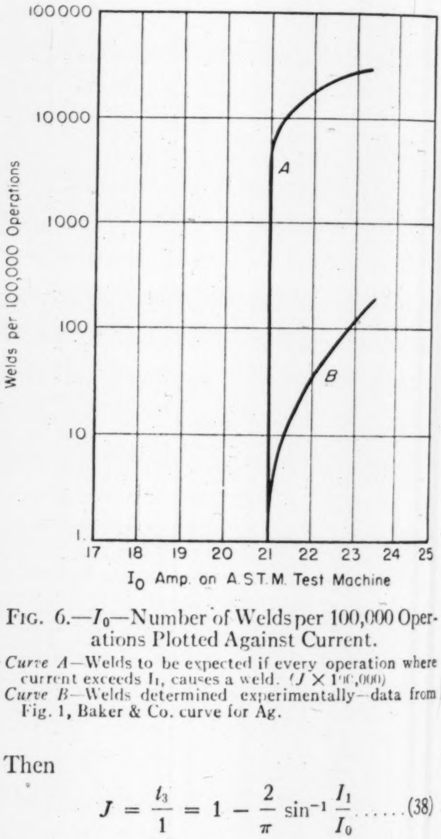

FIG. 6.—I_0—Number of Welds per 100,000 Operations Plotted Against Current.

Curve A—Welds to be expected if every operation where current exceeds I_1, causes a weld. ($J \times 100{,}000$)
Curve B—Welds determined experimentally—data from Fig. 1, Baker & Co. curve for Ag.

Then

$$J = \frac{t_3}{\dfrac{1}{4\nu}} = 1 - \frac{2}{\pi} \sin^{-1} \frac{I_1}{I_0} \quad \ldots\ldots(38)$$

Now, if a weld were to occur every time the contacts operate when the current exceeds $\sqrt{2}\,I_1$, there should be $J \times 100{,}000$ welds per 100,000 operations. This can be compared with the experimental data.

Figure 6 curve A shows $J \times 100{,}000$ plotted against I_0 and curve B shows the data for silver worked out by Baker & Co.

as shown in Fig. 1. The number of welds to be expected when the current exceeds I_1 increases very rapidly as shown by curve A. The fact that curve B is somewhat lower is because every operation where a weld is possible does not result in a weld due to the number of contact areas, oxide films, or other disturbing effects on the contact surfaces.

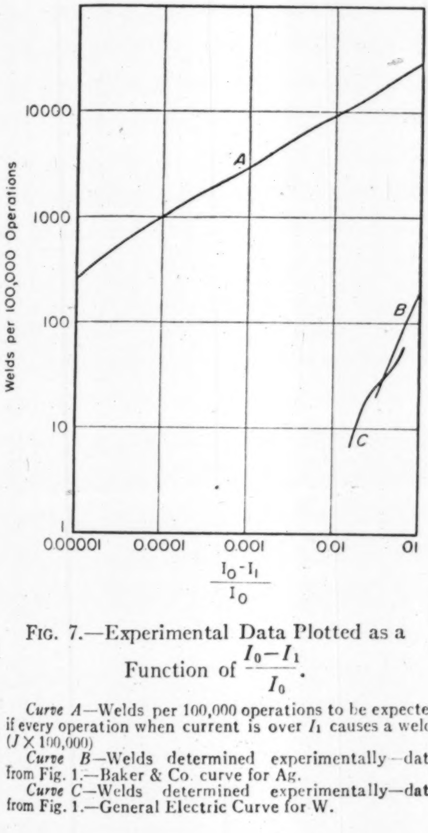

FIG. 7.—Experimental Data Plotted as a Function of $\frac{I_0-I_1}{I_0}$.

Curve A—Welds per 100,000 operations to be expected if every operation when current is over I_1 causes a weld. ($J \times 100{,}000$)

Curve B—Welds determined experimentally—data from Fig. 1.—Baker & Co. curve for Ag.

Curve C—Welds determined experimentally—data from Fig. 1.—General Electric Curve for W.

A somewhat different picture of the situation is shown in Fig. 7 in which $J \times 100{,}000$ and the experimental data from Fig. 1 are plotted as a function of $\frac{I_0 - I_1}{I_0}$. Curve A gives the number of welds to be expected if the contacts weld on every operation for which the current exceeds I_1. Curves B and C are the data taken from Fig. 1. It can be seen that these curves vary in position on the log-log scale by a factor between 200 and 2000. This means that welds occur on only one out of every 200 to 2000 of the operations on which a weld is possible.

One other factor to be considered is the sequence of welding. In the discussion to this point there has been no consideration of whether the weld could take place on opening or closing, or both. There is nothing in the calculations which indicates which is required and there is no direct experimental evidence to prove the point one way or another at this time. This point may be investigated on the machine without too much difficulty if it is found desirable to do so.

In any case, however, the values of J calculated above are correct if welding takes place on either opening or closing but not on both. If welding can take place on both, this means that there are two possibilities for a weld on each operation and the values of J should be doubled. It also means that welds occur only once out of every 400 to 4000 of the operations on which welds may be expected because of the circuit conditions.

It is possible to do little more than speculate on this part of the problem since the points on the experimental curves are subject to considerable variation and accurate data would be very difficult to secure. This discussion does little more than indicate that welding does not take place every time the circuit conditions are correct, and that the welds take place on the order of one out of every 1000 times the circuit conditions would permit. However, it does point out why the curves are so steep and why the limiting current is a factor which is subject to reasonably accurate measurement on a properly adjusted machine.

HARDNESS CONVERSION CHART FOR NICKEL AND HIGH-NICKEL ALLOYS*

By F. P. Huston, Jr.[1]

Synopsis

A correlation technique is described for hardness data. A procedure for classifying such data is given and linear relationships are established and least squares equations calculated from the classified data. A hardness conversion chart for nickel and high-nickel alloys, established by the above method, is presented. This chart is considered valid for the range of hardnesses normally encountered on such materials.

A technique for obtaining a valid correlation from a set of data covering measurements on test specimens is presented by means of a description of its application in establishing a hardness correlation for nickel and high-nickel alloys. The details of each phase of the correlation will not be given but can be obtained from references in the bibliography.

The present work consists of two separate, but interrelated, treatments; the first, the refinement or classification of a mass of hardness data; and the second, the use of the refined data in establishing linear relationships and both least squares and empirical equations.

Data

The data used in this correlation were of two types, namely, those obtained from hardness readings on a set of specimens and those obtained from a mass of both specific and general conversions established for the high-nickel alloys over a period of approximately 15 years, in the Bayonne and Huntington laboratories of The International Nickel Co. Inc., and the Wilson Mechanical Instrument Co. Laboratory. All the data were obtained by laboratory measurement and were intended for use in development, conversion, or production control work.

The major portion was obtained from a set of 51 specimens, finished to mill standards for hardness testing, covering the entire range of the high-nickel alloys from the softest to approximately the hardest with most of the intermediate forms represented. The samples were of homogeneous structure and no attempt was made to classify them according to grain size, which increases the universality of the correlation. From three to seven readings were taken of Vickers $\frac{2}{3}$–5 kg.,[2] Vickers $\frac{2}{3}$–30 kg. and fourteen Rockwell scales, including the superficial Rockwell, on each specimen.

In addition to the data obtained from the specimens, those available in the file were on solid solution and age-hardening

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Works Laboratory, The International Nickel Co., Inc., Huntington, W. Va.

[2] Vickers diamond pyramid, ⅔ objective and 5 kg. load.

alloys in rod, strip, sheet, tubing, and special forms. These materials had also been subjected to various thermal treatments. The alloys were:

1. Nickel,
2. Nickel-copper,
3. Nickel-copper-aluminum,
4. Nickel-chromium-iron, and
5. Nickel-aluminum-silicon.

It was thought that a representative sample was available and that a precise correlation should yield a useful chart relationship for the high-nickel alloys.

CLASSIFICATION OF DATA

The mass of hardness data collected over a period of years was not considered suitable for classification since these data, in most cases, indicated averages rather than individual readings. A method of classifying the data according to their reliability was developed and

TABLE I.—PRECISION MEASURES.[a]

$\sigma = \sqrt{\frac{\Sigma x^2}{n}}$	P.E. $= 0.6745 \sqrt{\frac{\Sigma x^2}{n}}$		
	Bessel's Formula	Peter's Formula	Approx. of Peter's Formula
σ'Est. $= \sqrt{\frac{x^2}{n-1}}$	P.E.' Est. $= 0.6745 \sqrt{\frac{\Sigma x^2}{n-1}}$	P.E. $= 0.8454\ \delta$	a.d. $= \frac{\Sigma x}{n}$
$\sigma_M = \frac{\sigma' \text{Est.}}{\sqrt{n}}$	P.E.$_M = \frac{\text{P.E.}' \text{Est.}}{\sqrt{n}}$	P.E.$_M = \frac{\text{P.E.}}{\sqrt{n}}$	A.D. $= \frac{\text{a.d.}}{\sqrt{n}}$
% Precision of Mean....	$\% = \frac{\text{P.E.}_M}{M} \times 100$	$\% = \frac{\text{P.E.}_M}{M} \times 100$	$\% = \frac{\text{A.D.}}{M} \times 100$

where:

σ = Standard deviation of sample.
σ'Est. = Estimated standard deviation of universe.
σ_M = Standard error of mean universe.
P.E. = Probable error.
δ and a.d. = Average deviation.
A.D. = Average deviation of the universe of means of samples of size n.
x = Absolute value of deviation from arithmetic mean, regardless of sign.
M = Arithmetic mean of series of observations.
Σ = Summation of series of observations.
n = Number of observations.

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[a] The derivation chart for the precision measures is self-explanatory. The first two columns are the fundamental measures of standard deviation and probable error respectively for more than ten observations. The third column gives the equivalent forms of Peter's formula, which is an approximation of Bessel's formula. The fourth column gives the approximation formulae commonly used and which are good only for from three to eight observations. The approximation is along each line. If the number of measurements is few and if there are no very discordant values, the average deviation can be used as a precision measure. The average deviation is smaller than the probable error calculated by Peter's formula for less than three observations, while for greater than eight observations the average deviation is larger than the probable error calculated by Peter's formula. For very few measurements, the discrepancies are of the order of magnitude of the uncertainties in the values of the probable error calculated by Peter's formula.

The use of the approximation formulas is quite limited and not generally recommended. They can be applied only when they are statistically "good enough" for the type of data involved.

Example.—Using Specimen H4 and Approximation of Peter's Formula

1. Calculation of arithmetic mean of a series of observations:—Rockwell C readings—31.0, 31.1, 31.2, 32.5, 30.5 and 31.1
   Σ = Summation of readings = 187.4
   n = Number of readings = 6
   M = Arithmetic mean $= \frac{187.4}{6} = 31.2$
2. Calculation of deviation from arithmetic mean, regardless of sign:—
   $\Sigma x = (31.2 - 31.0) + (31.2 - 31.1) + (31.2 - 31.2) + (32.5 - 31.2) + (32.5 - 31.2) + (31.2 - 30.5) + (31.2 - 31.1) = 2.4$
3. Calculation of average deviation:—
   a.d. $= \frac{\Sigma x}{n} = \frac{2.4}{6} = \pm 0.4$
4. Calculation of average deviation of the universe of means of samples of size n.
   A.D. $= \pm \frac{\text{a.d.}}{\sqrt{n}} = \frac{0.4}{\sqrt{6}} = \pm 0.16$
5. Calculation of per cent precision:—
   % Precision $= \pm \frac{\text{A.D.}}{M} \times 100 = \pm \frac{0.16}{31.2} \times 100 = \pm 0.5\%$

NOTE.—For the example shown above the mean can be expected 50 per cent of the time to l within the band of Rockwell C value of 31.2 ± 0.5 per cent.

TABLE II.—HARDNESS DATA

Number	Specimen	Metal*	VHN 5-kg. Load Average	± a.d.	± A.D.	Precision, per cent	VHN 30-kg. Load Average	± a.d.	± A.D.	Precision, per cent	R_b Average	± a.d.	± A.D.	Precision, per cent	R_e Average	± a.d.	± A.D.	Precision, per cent	R_f Average	± a.d.	± A.D.	Precision, per cent	R_d Average
1...	U	A-Ni	82.6	2.7	0.8	1.0	79.7	0.8	0.5	0.6	37.4	0.6	0.3	1.0	−46.3	0.6	0.4	0.8	75.0	0.7	0.4	0.5	...
2...	O		85.1	0.8	0.4	0.5	83.7	0.9	0.5	0.6	38.3	0.3	0.2	0.4	−44.8	0.2	0.1	0.3	74.6	1.8	1.0	1.3	...
3...	R		85.7	2.3	0.9	1.1	82.7	3.2	1.9	2.3[a]	38.7	0.4	0.3	0.6	−48.2	0.8	0.5	1.0	...	...	...	...	...
4...	T		84.6	2.7	1.6	1.8	81.8	0.7	0.4	0.5	39.2	0.5	0.3	0.7	−45.3	0.3	0.2	0.4	79.0	0.5	0.3	0.4	...
5...	V		79.0	0.8	0.3	0.4	81.3	1.1	0.6	0.8	40.1	0.9	0.5	1.0	−47.5	0.5	0.3	0.6	78.3	1.1	0.6	0.8	...
6...	Q		86.0	1.7	0.6	0.8	85.2	0.7	0.4	0.5	43.0	0.1	0.1	0.1	−41.2	0.4	0.2	0.5	78.0	2.0	1.2	1.5	...
7...	P		103.0	1.7	0.6	0.6	102.0	4.0	1.5	1.5	54.4	1.6	0.9	1.7	−29.6	0.7	0.4	1.4	88.1	0.2	0.1	0.1	...
8...	S		102.0	3.0	1.5	1.5	100.3	1.6	0.9	0.9	55.5	1.2	0.7	1.2	−29.5	1.0	0.6	2.0	87.7	0.2	0.1	0.1	2.4
9...	17	B-M	117.0	1.7	1.0	0.8	106.0	1.3	0.8	0.7	56.3	0.7	0.4	0.7	−29.3	0.6	0.4	1.2	88.3	0.7	0.4	0.5	2.9
10...	8		118.0	2.6	1.0	0.8	112.0	0.3	0.2	0.2	61.5	0.3	0.2	0.3	−23.1	0.1	0.02	0.1	90.8	0.7	0.4	0.4	7.7
11...	11		125.0	1.5	0.6	0.5	123.0	1.7	1.0	0.8	66.8	0.3	0.2	0.3	−17.1	0.4	0.2	1.2	94.3	0.4	0.2	0.2	10.9
12...	7		125.0	1.0	0.6	0.5	122.0	0.7	0.4	0.3	67.7	0.6	0.3	0.5	−15.8	0.4	0.2	1.3	95.0	0.3	0.2	0.2	12.6
13...	A		123.0	1.7	0.7	0.6	121.0	1.3	0.8	0.6	68.5	0.2	0.1	0.2	−14.5	0.5	0.3	2.0	94.7	0.2	0.1	0.1	13.8
14...	14		129.0	2.3	1.3	1.0	124.0	2.3	1.3	1.1	69.6	0.2	0.1	0.1	−14.4	0.4	0.2	1.4	95.6	0.4	0.2	0.2	14.4
15...	B		120.0	2.8	1.1	0.9	124.0	1.0	0.6	0.5	70.1	0.7	0.4	0.5	−12.7	0.5	0.2	1.6	95.8	0.2	0.1	0.1	...
16...	Z	NX	154.0	6.0	1.7	1.1	140.4	3.8	1.4	1.0	71.8	1.2	0.7	0.9	−12.2	0.4	0.1	1.2	97.5	0.3	0.2	0.2	15.2
17...	21		153.0	2.5	0.7	0.4	152.0	0.3	0.2	0.1	76.5	0.4	0.2	0.3	−6.6	0.3	0.2	2.9[a]	99.3	0.3	0.2	0.2	19.5
18...	Y		160.0	6.4	2.4	1.5	154.0	0.3	0.2	0.1	78.1	0.5	0.3	0.4	−4.5	0.7	0.2	5.0[a]	100.5	0.3	0.2	0.2	20.5
19...	X		166.0	5.0	2.1	1.2	...	...	...	...	75.1	1.3	0.8	1.0	−7.1	1.1	0.4	4.7[a]	98.0	1.3	0.5	0.5	18.1
20...	W		177.0	4.4	1.0	0.6	178.0	3.7	2.1	1.2	86.1	0.2	0.1	0.1	3.9	0.2	0.1	1.9	104.5	0.3	0.2	0.2	28.3
21...	23		165.0	4.2	1.3	0.8	161.5	2.7	1.1	0.7	82.0	0.3	0.2	0.2	−0.6	0.4	0.1	2.2[a]	102.6	0.5	0.2	0.2	25.5
22...	22		205.0	4.3	1.2	0.6	197.0	0.3	0.2	0.1	89.7	0.3	0.1	0.2	8.2	0.2	0.1	0.8	107.2	0.2	0.1	0.1	32.2
23...	4	B-M	206.0	2.0	1.2	0.6	209.0	5.7	3.3	1.6	94.1	0.4	0.2	0.2	13.5	0.8	0.2	1.8	109.9	0.5	0.3	0.3	35.1
24...	1		232.0	2.7	1.6	0.7	238.0	3.3	1.3	0.6	99.1	0.2	0.1	0.1	20.2	0.3	0.1	0.6	112.6	0.2	0.1	0.1	40.2
25...	E		249.0	2.3	0.7	0.3	251.8	5.3	2.0	0.8	100.2	0.1	0.1	0.1	22.6	0.5	0.2	0.8	112.8	1.7	1.0	0.9	42.1
26...	L		246.0	4.4	1.7	0.7	258.0	5.1	2.0	0.8	102.0	0.1	0.1	0.1	25.0	0.4	0.2	0.7	115.7	0.8	0.5	0.4	43.8
27...	Z_1		248.0	3.1	0.8	0.3	255.0	1.3	0.8	0.3	102.0	0.6	0.3	0.3	23.3	0.1	0.1	0.4	114.0	0.1	0.1	0.1	42.9
28...	N		272.0	4.0	2.3	0.9	271.0	1.0	0.6	0.2	103.7	0.1	0.1	0.1	26.4	0.3	0.2	0.8	115.7	0.8	0.5	0.4	45.0
29...	J_2		260.0	1.7	0.6	0.2	269.0	3.7	2.1	0.8	104.0	0.2	0.1	0.1	26.6	0.2	0.1	0.4	116.0	1.0	0.6	0.5	45.1
30...	D	K-M	287.0	12.3	3.4	1.2	280.0	11.3	6.5	2.3[a]	106.6	0.3	0.2	0.2	30.7	0.3	0.1	0.4	117.0	0.1	0.1	0.1	48.0
31...	H_4		291.5	8.3	3.4	1.2	298.5	2.1	0.9	0.3	106.9	0.1	0.1	0.1	31.2	0.4	0.2	0.5	117.2	0.2	0.1	0.1	48.1
32...	K_1		319.0	4.7	1.4	0.4	324.0	2.3	1.3	0.4	106.9	0.3	0.2	0.2	32.8	0.2	0.1	0.3	118.2	0.5	0.3	0.3	50.0
33...	H_8		285.0	6.2	2.1	0.7	300.4	4.4	1.8	0.6	107.3	0.2	0.1	0.1	31.2	0.3	0.1	0.4	116.3	0.2	0.1	0.1	48.3
34...	I_4		303.0	6.0	2.0	0.7	320.0	4.0	1.3	0.4	107.2	0.2	0.1	0.1	33.2	0.4	0.2	0.6	118.0	0.0	0.0	0.0	50.0
35...	E_1		331.0	0.3	0.1	0.1	355.0	25.0	11.0	3.2[a]	107.5	0.0	0.0	0.0	34.7	0.2	0.1	0.3	118.2	0.9	0.5	0.4	51.2
36...	C		317.0	12.0	4.9	1.6	321.0	0.3	0.2	0.1	107.6	0.3	0.2	0.2	33.2	0.2	0.1	0.2	117.8	1.0	0.6	0.5	50.0
37...	20	K′-M	305.0	8.0	2.2	0.7	310.3	5.5	2.3	0.7	107.7	0.4	0.2	0.2	32.2	0.4	0.1	0.4	117.8	0.2	0.1	0.1	49.0
38...	G_2		377.0	7.7	2.1	0.6	374.0	11.3	4.6	1.2	112.1	0.3	0.2	0.2	40.0	0.3	0.1	0.3	119.9	0.1	0.1	0.1	55.2
39...	D_1		361.0	9.0	3.7	1.0	381.5	4.0	1.6	0.4	112.3	0.2	0.1	0.1	41.7	0.9	0.4	0.9	120.0	0.1	0.1	0.1	55.8
40...	I_3		426.0	4.8	2.2	0.5	398.0	5.0	2.9	0.7	112.7	0.4	0.3	0.2	42.5	0.3	0.1	0.3	120.7	0.3	0.2	0.2	57.4
41...	M		399.0	4.3	1.6	0.4	407.0	8.3	4.8	1.2	112.7	0.1	0.1	0.1	43.2	0.5	0.2	0.4	120.8	0.2	0.1	0.1	57.1
42...	C_1		402.0	6.3	2.1	0.5	417.7	5.0	1.9	0.5	113.0	0.2	0.1	0.1	42.9	0.2	0.1	0.2	121.2	0.2	0.1	0.1	57.8
43...	H_7		395.0	1.7	1.0	0.3	400.0	8.7	5.0	1.3	113.0	0.1	0.1	0.1	41.9	0.1	0.1	0.2	120.0	0.1	0.1	0.1	56.7
44...	5	K-M	412.0	6.0	2.0	0.5	425.0	3.7	2.1	0.5	113.9	0.3	0.2	0.2	42.8	0.2	0.1	0.2	121.2	0.6	0.3	0.2	58.0
45...	H_3		395.0	6.3	3.6	0.9	395.0	1.3	0.8	0.2	112.9	0.9	0.5	0.5	41.4	0.3	0.2	0.5	120.0	0.1	0.1	0.1	56.6
46...	1B	B-M	156.0	0.6	0.2	0.1	156.0	1.0	0.6	0.4	80.8	0.5	0.3	0.3	...	...	...	...	...	...	...	...	...
47...	2B		158.0	1.0	0.6	0.4	150.0	2.0	1.0	0.8	80.8	0.6	0.3	0.4	...	...	...	...	...	...	...	...	...
48...	3B		147.0	1.0	0.6	0.4	148.0	1.0	0.6	0.4	77.4	0.7	0.4	0.5	...	...	...	...	...	...	...	...	...
49...	4B		137.0	2.0	1.0	0.8	139.0	3.0	2.0	1.2	76.8	0.4	0.2	0.3	...	...	...	...	...	...	...	...	...
50...	5B		135.0	2.0	1.0	0.8	134.0	1.0	0.6	0.4	79.9	0.6	0.3	0.5	...	...	...	...	...	...	...	...	...
51...	6B		133.0	0.3	0.2	0.2	133.0	2.0	1.0	0.6	71.2	0.4	0.2	0.3	...	...	...	...	...	...	...	...	...

[a] Eliminated by 2 per cent criterion.

* A-Ni Nickel
B-M Monel (Reg. U. S. Pat. Off.)
NX Inconel (Reg. U. S. Pat. Off.)
K-M "K" Monel (Reg. U. S. Pat. Off.)

AND PRECISION MEASURES.

± a.d.	± A.D.	Precision, per cent	R_e Average	± a.d.	± A.D.	Precision, per cent	R_k Average	± a.d.	± A.D.	Precision, per cent	15*N* Average	± a.d.	± A.D.	Precision, per cent	30*N* Average	± a.d.	± A.D.	Precision, per cent	45*N* Average	± a.d.	± A.D.	Precision, per cent	R_a Average	± a.d.	± A.D.	Precision, per cent
...	...	...	75.3	0.4	0.2	0.3	43.7	1.2	0.6	1.4	...	...	...	...	...	...	...	...	...	...	...	...	28.8	0.2	0.1	0.5
...	...	...	77.5	0.0	0.0	0.0	51.8	0.2	0.1	0.2	...	...	...	...	...	...	...	...	...	...	...	...	29.5	0.3	0.2	0.7
...	...	...	75.5	0.3	0.2	0.3	46.4	1.5	0.7	1.5	...	...	...	...	...	...	...	...	...	...	...	...	27.2	0.1	0.1	0.2
...	...	...	76.8	0.4	0.2	0.3	50.8	1.0	0.4	0.8	...	...	...	...	...	...	...	...	...	...	...	...	29.0	0.0	0.0	0.0
...	...	...	75.8	0.6	0.3	0.4	49.0	0.0	0.0	0.0	...	...	...	...	...	...	...	...	...	...	...	...	27.8	0.6	0.3	1.2
...	...	...	79.0	0.3	0.2	0.2	48.2	0.9	0.5	1.1	...	...	...	...	...	...	...	...	...	...	...	...	30.7	0.4	0.2	0.7
...	...	...	86.9	0.6	0.3	0.4	60.6	1.1	0.6	0.9	...	...	...	...	...	...	...	...	...	...	...	...	35.8	0.6	0.3	0.9
1.1	0.5	20.0[a]	85.3	0.2	0.1	0.2	60.4	1.4	0.6	1.0	...	...	...	...	...	...	...	...	...	...	...	...	35.8	0.2	0.1	0.4
0.9	0.4	15.0[a]	86.3	0.4	0.2	0.3	65.2	0.8	0.4	0.7	...	...	...	...	...	...	...	...	...	...	...	...	36.2	0.2	0.1	0.4
0.4	0.2	2.6[a]	89.7	0.2	0.1	0.1	70.5	1.0	0.6	0.8	...	...	...	...	...	...	...	...	...	...	...	...	39.2	0.2	0.1	0.3
0.5	0.3	2.4[a]	91.7	0.4	0.2	0.3	74.4	0.6	0.3	0.4	...	...	...	...	...	...	...	...	...	...	...	...	41.5	0.3	0.2	0.5
0.1	0.1	0.6	93.5	0.0	0.0	0.0	77.2	0.2	0.1	0.2	...	...	...	...	...	...	...	...	...	...	...	...	42.5	0.3	0.2	0.5
0.1	0.1	0.4	94.7	0.2	0.1	0.1	76.7	0.4	0.2	0.3	...	...	...	...	...	...	...	...	...	...	...	...	43.0	0.0	0.0	0.0
0.7	0.3	2.3[a]	94.8	0.2	0.1	0.1	76.7	0.2	0.1	0.2	...	...	...	...	...	...	...	...	...	...	...	...	43.8	0.2	0.1	0.2
...	...	...	95.0	0.0	0.0	0.0	77.0	0.0	0.0	0.0	...	...	...	...	...	...	...	...	...	...	...	...	44.0	0.0	0.0	0.0
0.2	0.1	0.9	94.7	0.2	0.1	0.1	77.2	0.2	0.1	0.2	55.9	0.1	0.1	0.1	18.1	0.2	0.1	0.7	−14.5	0.8	0.3	2.3[a]	43.7	0.2	0.1	0.3
0.3	0.2	1.0	99.7	0.2	0.1	0.1	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	47.3	0.2	0.1	0.3
0.3	0.2	0.9	100.6	0.0	0.0	0.0	83.1	0.9	0.4	0.5	58.6	0.4	0.2	0.4	21.9	0.8	0.5	2.0	−7.4	0.5	0.2	2.9[a]	47.5	0.3	0.2	0.4
0.4	0.2	1.3	99.4	0.3	0.2	0.2	80.3	0.4	0.2	0.3	58.5	0.5	0.3	0.5	18.4	0.3	0.2	0.9	−11.3	0.4	0.2	2.2[a]	45.7	0.9	0.5	1.1
0.4	0.2	0.7	105.6	0.2	0.1	0.1	90.0	0.0	0.0	0.0	62.1	0.3	0.2	0.3	28.1	0.9	0.5	1.9	2.0	0.0	0.0	0.0	52.2	0.2	0.1	0.3
0.3	0.2	0.8	103.2	0.2	0.1	0.1	86.3	0.2	0.1	0.2	60.0	0.0	0.0	0.0	23.8	0.4	0.2	0.9	−4.4	0.7	0.3	7.4[a]	49.7	0.2	0.1	0.3
0.6	0.3	1.1	108.5	0.2	0.1	0.1	93.6	0.2	0.1	0.1	65.0	0.0	0.0	0.0	32.2	0.3	0.1	0.4	5.6	0.3	0.1	2.6[a]	54.8	0.2	0.1	0.2
0.6	0.3	1.0	112.1	0.2	0.1	0.1	98.2	0.6	0.3	0.3	67.0	0.3	0.2	0.3	36.0	0.5	0.3	0.8	11.2	0.7	0.4	3.9[a]	57.2	0.2	0.1	0.2
0.4	0.2	0.6	...	...	...	...	106.0	0.3	0.2	0.2	69.1	0.5	0.3	0.4	40.3	0.2	0.1	0.3	18.2	0.3	0.2	0.9	60.0	0.0	0.0	0.0
0.2	0.1	0.2	...	...	...	...	107.4	0.1	0.1	0.1	...	...	...	...	...	...	...	...	...	...	...	...	61.6	0.1	0.1	0.1
0.4	0.2	0.5	...	...	...	...	109.7	0.2	0.1	0.1	...	...	...	...	...	...	...	...	...	...	...	...	62.5	0.3	0.2	0.3
0.1	0.1	0.1	...	...	...	...	109.0	0.0	0.0	0.0	...	...	...	...	...	...	...	...	...	...	...	...	62.0	0.0	0.0	0.0
0.1	0.02	0.04	...	...	...	...	110.4	0.1	0.1	0.1	...	...	...	...	...	...	...	...	...	...	...	...	63.3	0.1	0.1	0.1
0.1	0.1	0.1	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	63.3	0.1	0.1	0.1
0.03	0.02	0.03	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	65.4	0.3	0.2	0.2
0.1	0.05	0.1	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	65.9	0.1	0.1	0.1
0.0	0.0	0.0	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	67.2	0.2	0.1	0.2
0.2	0.1	0.2	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	65.8	0.2	0.1	0.2
0.1	0.1	0.1	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	67.0	0.0	0.0	0.0
0.3	0.2	0.4	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	68.0	0.0	0.0	0.0
0.0	0.0	0.0	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	67.0	0.0	0.0	0.0
0.2	0.1	0.2	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	70.6	0.2	0.1	0.2
0.3	0.1	0.2	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	71.0	0.0	0.0	0.0
0.1	0.1	0.1	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	72.1	0.3	0.2	0.2
0.3	0.2	0.3	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	71.9	0.1	0.1	0.1
0.1	0.1	0.1	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	72.2	0.2	0.1	0.2
0.03	0.02	0.03	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	71.5	0.0	0.0	0.0
0.1	0.1	0.1	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	72.2	0.2	0.1	0.2
0.03	0.02	0.03	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	71.3	0.2	0.1	0.1
0.6	0.4	0.6	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...

applied. Approximation formulas for which derivations are given in Table I, were used. The data given in Table II were obtained by the author on the set of 51 specimens. A measure of ± 2 per cent was chosen as the standard. In applying this arbitrary standard Rockwell B readings in the neighborhood of 100 R_b must be within $\pm$ 2 R_b and those in the neighborhood of 50 R_b must be within $\pm$ 1 R_b. Least squares and empirical equations were calculated from these data and the curves of the equations so developed were plotted. When the data from the file were plotted with the equations it was found that practi-

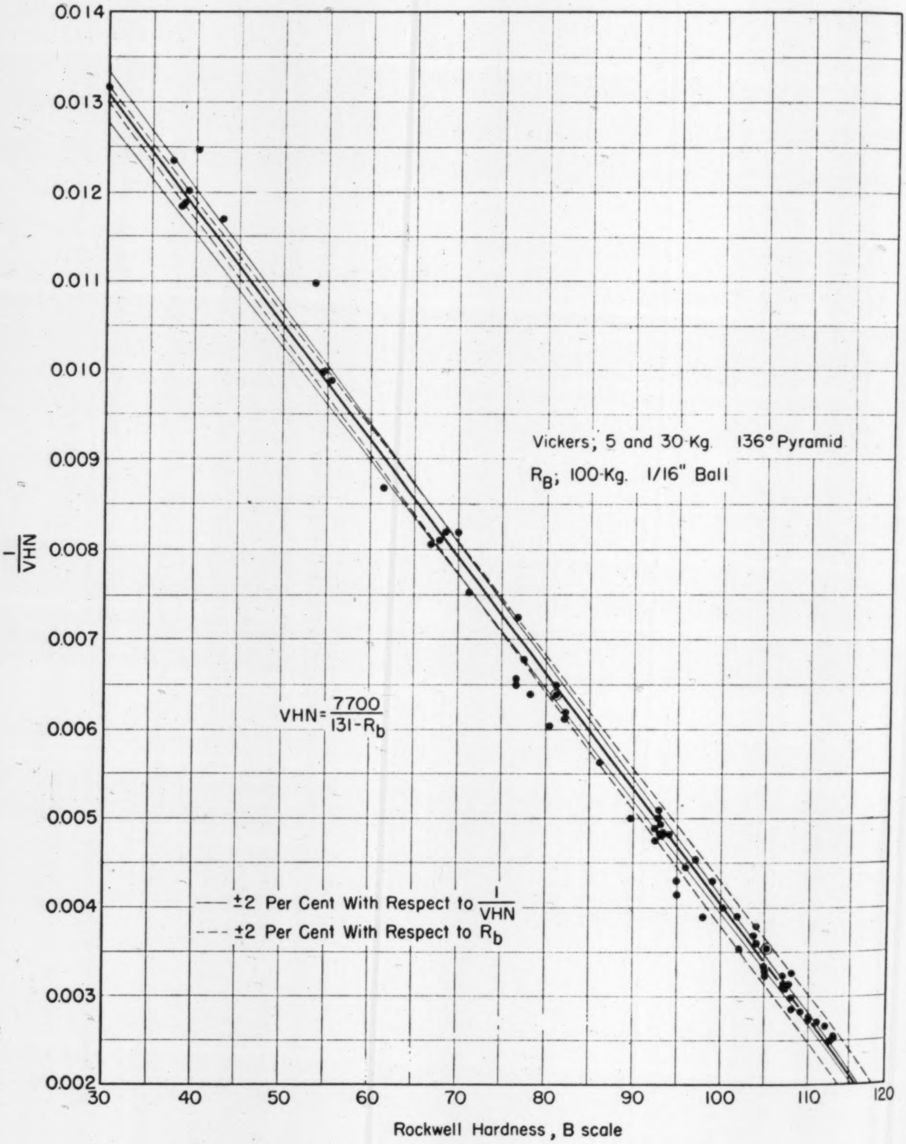

Fig. 1.—Reciprocal Vickers Hardness *versus* Rockwell B.

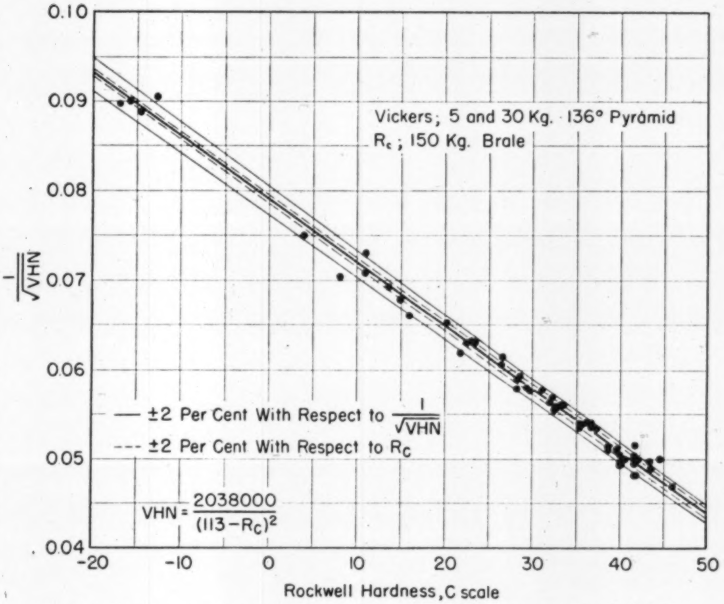

Fig. 2.—Reciprocal Square Root Vickers Hardness *versus* Rockwell C.

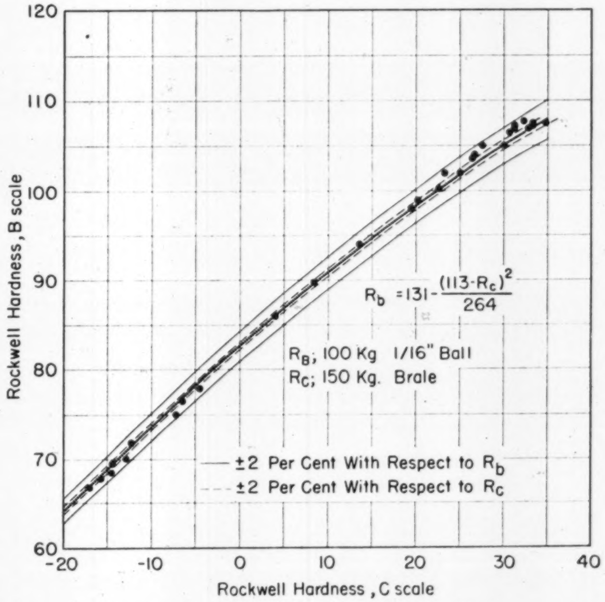

Fig. 3.—Rockwell B *versus* Rockwell C.

cally all points fell within the band established as ± 2 per cent of the mean.

The line of best fit, ± 2 per cent bands (one band with respect to the ordinate and the other band with respect to the abscissa), and the points used are shown in Figs. 1, 2, and 3 for the $\frac{1}{\text{VHN}}$ *versus* R_b, $\frac{1}{\sqrt{\text{VHN}}}$ *versus* R_c, and R_b *versus* R_c equations.

EQUATIONS

The data were plotted so that linear functions were obtained for VHN *versus* R_b and VHN *versus* R_c. Least squares equations were calculated for the VHN *versus* R_b, VHN *versus* R_c, and R_b *versus* R_c relations.

S. Petrenko (**4, 5**)[3] showed that R_c varies linearly as the reciprocal of the square root of the Brinell number, and R_b varies linearly as the reciprocal of the Brinell number. In the present investigation, it was found that the same relations can be used for Rockwell *versus* Vickers.

The methods of statistical analysis (**1, 2, 6**) were used in obtaining the relationship between Vickers, Rockwell B, and Rockwell C. Least squares equations were calculated for $\frac{1}{\text{VHN}}$ *versus* R_b, $\frac{1}{\sqrt{\text{VHN}}}$ *versus* R_c and R_b *versus* R_c. An equation of the form $Y = mX + b$ was used to calculate the Vickers, R_b and R_c relationships. The final equations were:

$$\text{VHN} = \frac{7700}{131 - R_b} \quad \ldots\ldots\ldots (1)$$[4]

and

$$\text{VHN} = \frac{2{,}038{,}000}{(113 - R_c)^2} \quad \ldots\ldots. (2)$$[5]

The adjusted coefficients of correlation for the above equations were 0.991 and 0.996, respectively.

It was observed that the relationship between R_b and R_c, the ball and the "Brale," was parabolic, or could be represented by a parabola, and an equation of the form $Y = a + bX + cX^2$ was used. This resulted in a R_b *versus* R_c relationship of Eq. 6; namely,

$$R_b = 131 - \frac{(113 - R_c)^2}{264}$$

The equations for the relationships between R_b, R_c, and Brinell were determined empirically by plotting $\frac{1}{\text{BHN}}$ *versus* R_b and $\frac{1}{\sqrt{\text{BHN}}}$ *versus* R_c from a comprehensive series of conversions in the file and obtaining the equations therefrom. Thus, the empirical equations are:

$$\text{BHN} = \frac{7800}{132.3 - R_b} \quad \ldots\ldots\ldots (3)$$

and

$$\text{BHN} = \frac{2{,}075{,}000}{(115.5 - R_c)^2} \quad \ldots\ldots. (4)$$

The assumption of perfect compatability among all the data involved in the derivation of the chart necessitates that a mathematical consistency exist between the various equations. In order to achieve this, certain minor adjustments had to be made in the constants of the R_b *versus* R_c relations derived from the several sources. It was found that the R_b:VHN and R_c:VHN equations gave rise to a slightly different set of constants in an equation of the same form than that obtained from the corresponding R_b:BHN and R_c:BHN equations. Furthermore, least squares studies of the cases in which both R_b and R_c tests were made on the same specimens produced another set of constants.

It was decided that the best solution to this situation was a compromise equation, Eq. 6, in which the constants were adjusted arbitrarily. It must be understood clearly that these differences were very small indeed.

In arriving at the VHN:BHN relation the least squares R_c:VHN and the R_b:BHN obtained from the file were considered valid. The desirability of retaining mathematical consistency between the least squares VHN:R_b:R_c data and the BHN:R_b:R_c data from the file was recognized, and for that reason greater weight was given to those data from which least squares equations could be derived. This necessitated an adjustment in the R_c:BHN equation from the file data to make it consistent with the R_b:BHN and R_c:VHN equations and the R_b:R_c Eq. 6 discussed above. The R_b:BHN relationship was considered valid because of the distribution and number of data. The nonlinear VHN:BHN equation so obtained is:

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[3] The boldface numbers in parentheses refer to the references appended to this paper, see p. 780.

[4] Original Formula:
$Y = 0.01703 - 0.0001304\ X$, where
Y = Reciprocal of Vickers hardness reading, and
X = Rockwell hardness reading, B scale.

[5] Original Formula:
$Y = 0.07885 - 0.0007005\ X$, where
Y = Reciprocal of the square root of the Vickers hardness reading, and
X = Rockwell hardness reading, C scale.

$$\text{BHN} = \frac{6000\ \text{VHN}}{5925 + \text{VHN}} \quad \ldots\ldots(5)$$

As the relationship between the ball and the "Brale" is parabolic in form, and that between one ball scale and another, and between one "Brale" scale and another are linear, all the ball scales were plotted against the R_b scale, and all "Brale" scales were plotted against the R_c scale.

If the assumption is made that Rockwell B is linear over a particular range, based on considerations of depth of indentation and possible deformation of the ball, and any other Rockwell ball scale is plotted *versus* the Rockwell B readings and a departure from linearity is observed, then it may be considered that the nonlinearity is produced by the other scale. The same analysis will hold for all "Brale" scales when the Rockwell C scale is used as a standard. The range of linearity of all the Rockwell scales can be established by the above method.

Empirical equations were obtained for each relationship over the range considered valid. Thus, the best equations for VHN *versus* R_b, VHN *versus* R_c, and R_b *versus* R_c; and the empirical equations for all the Rockwell scales *versus* either R_b or R_c are established.

These equations were used as the basis for the chart which is given in this paper. It will be noted that ranges are given with the equations; for example, from 30 R_b to 106 R_b. This means that the equation was derived from significant data which extended from 30 R_b to 106 R_b.

The equations, ranges over which they hold, penetrator used, and the number of the equation are given:

VHN *versus* R_b.—Range, 30 R_b to 108 R_b, 136 deg. Pyramid and 1/16-in. diameter.

$$\text{VHN} = \frac{7700}{131 - R_b} \quad \ldots\ldots\ldots(1)$$

VHN *versus* R_c.—Range, −20 R_c to 50 R_c, 136 deg. Pyramid and Brale

$$\text{VHN} = \frac{2{,}038{,}000}{(113 - R_c)^2} \quad \ldots\ldots\ldots(2)$$

BHN *versus* R_b.—Range, 30 R_b to 108 R_b, 10-mm. diameter and 1/16-in. diameter

$$\text{BHN} = \frac{7800}{132.3 - R_b} \quad \ldots\ldots\ldots(3)$$

BHN *versus* R_c.—Range, 1 R_c to 50 R_c, 10-mm. diameter and Brale

$$\text{BHN} = \frac{2{,}075{,}000}{(115.5 - R_c)^2} \quad \ldots\ldots\ldots(4)$$

VHN *versus* BHN.—Range, 77 to 513 VHN, 136 deg. Pyramid and 10-mm. diameter

$$\left.\begin{aligned} \text{VHN} &= \frac{5925 - \text{BHN}}{6000 - \text{BHN}} \\ \text{BHN} &= \frac{6000\ \text{VHN}}{5925 + \text{VHN}} \end{aligned}\right\} \quad \ldots\ldots(5)$$

R_b *versus* R_c.—Range, −20 R_c to 108 R_b, 1/16-in. diameter and Brale

$$131 - R_b = \frac{(113 - R_c)^2}{264} \quad \ldots\ldots(6)$$

Empirical Equations:

R_b *versus* 15*T*.—Range, 30 R_b to 108 R_b, 1/16-in. diameter and 1/16-in. diameter

$$R_b = 2.82(15T) - 161.0 \quad \ldots\ldots\ldots\ldots(7)$$

R_b *versus* 30*T*.—Range, 30 R_b to 108 R_b, 1/16-in. diameter and 1/16-in. diameter

$$R_b = 1.47(30T) - 20.0 \quad \ldots\ldots\ldots\ldots(8)$$

R_b *versus* 45*T*.—Range, 30 R_b to 108 R_b, 1/16-in. diameter and 1/16-in. diameter

$$R_b = 1.01(45T) + 28.2$$

HARDNESS CONVERSION CHART FOR NICKEL AND HIGH-NICKEL ALLOYS.

Approximate relationships between hardness values.

Vickers Hardness Numbers VHN	Brinell Hardness Numbers BHN	ROCKWELL HARDNESS NUMBERS (Scales as indicated below)								"ROCKWELL-SUPERFICIAL" HARDNESS NUMBERS—(Scales as indicated below)					
		A	B	C	D	E	F	G	K	15-N	30-N	45-N	15-T	30-T	45-T
Diamond Pyramid Penetrator—1, 5, 10, 30-kg. Load; also 120-kg. Load with 2 mm. Ball Penetrator	Standard Type—3000-kg. Load with 10-mm. Ball Penetrator; also 500-kg. Load for Softer Metals	Diamond Cone "Brale" Penetrator, 60-kg. Load	1/16-in. Ball Penetrator 100-kg. Load	"Brale" Penetrator 150-kg. Load	"Brale" Penetrator 100-kg. Load	1/8-in. Ball Penetrator 100-kg. Load	1/16-in. Ball Penetrator 60-kg. Load	1/16-in. Ball Penetrator 150-kg. Load	1/8-in. Ball Penetrator 150-kg. Load	Sphero-Conical Diamond "N-Brale" Penetrator; 15-kg. Load	"N-Brale" Penetrator 30-kg. Load	"N-Brale" Penetrator 45-kg. Load	1/16-in. Ball Penetrator 15-kg. Load	1/16-in. Ball Penetrator 30-kg. Load	1/16-in. Ball Penetrator 45-kg. Load
513	479	75.5	....	50.0	63.0	....	....	....	....	85.5	68.0	54.5	....	....	....
481	450	74.5	....	48.0	61.5	....	....	....	....	84.5	66.5	52.5	....	....	....
452	425	73.5	....	46.0	60.0	....	....	....	....	83.5	64.5	50.0	....	....	....
427	403	72.5	....	44.0	58.5	....	....	....	....	82.5	63.0	47.5	....	....	....
404	382	71.5	....	42.0	57.0	....	....	....	....	81.5	61.0	45.5	....	....	....
382	363	70.5	....	40.0	55.5	....	....	....	....	80.5	59.5	43.0	....	....	....
362	346	69.5	....	38.0	54.0	....	....	....	....	79.5	58.0	41.0	....	....	....
344	329	68.5	....	36.0	52.5	....	....	....	....	78.5	56.0	38.5	....	....	....
326	313	67.5	....	34.0	50.5	....	....	....	....	77.5	54.5	36.0	....	....	....
309	298	66.5	*106*	32.0	49.5	....	*116.5*	94.0	....	76.5	52.5	34.0	*94.5*	*85.5*	*77.0*
285	275	64.5	*104*	28.5	46.5	....	*115.5*	91.0	....	75.0	49.5	30.0	*94.0*	*84.5*	*75.0*
266	258	63.0	*102*	25.5	44.5	....	*114.5*	87.5	....	73.5	47.0	26.5	*93.0*	*83.0*	*73.0*
248	241	61.5	100	22.5	42.0	....	*113.0*	84.5	....	72.0	44.5	23.0	92.5	81.5	71.0
234	228	60.5	98	20.0	40.0	....	*112.0*	81.5	....	70.5	42.0	20.0	92.0	80.5	69.0
220	215	59.0	96	*17.0*	*38.0*	....	*111.0*	78.5	100.0	*69.0*	*39.5*	*17.0*	91.0	79.0	67.0
209	204	57.5	94	*14.5*	*36.0*	....	*110.0*	75.5	98.0	*68.0*	*37.5*	*14.0*	90.5	77.5	65.0
198	194	56.5	92	*12.0*	*34.0*	....	*108.5*	72.0	96.5	*66.5*	*35.5*	*11.0*	89.5	76.0	63.0
188	184	55.0	90	*9.0*	*32.0*	*108.5*	*107.5*	69.0	94.5	*65.0*	*32.5*	*7.5*	89.0	75.0	61.0
179	176	53.5	88	*6.5*	*30.0*	*107.0*	*106.5*	65.5	93.0	*64.0*	*30.5*	*5.0*	88.0	73.5	59.5
171	168	52.5	86	*4.0*	*28.0*	*106.0*	*105.0*	62.5	91.0	*62.5*	*28.5*	*2.0*	87.5	72.0	57.5
164	161	51.5	84	*2.0*	*26.5*	*104.5*	*104.0*	59.5	89.0	*61.5*	*26.5*	*−0.5*	87.0	70.5	55.5
157	155	50.0	82	....	*24.5*	*103.0*	*103.0*	56.5	87.5	....	....	....	86.0	69.5	53.5
151	149	49.0	80	....	*22.5*	*102.0*	*101.5*	53.0	85.5	....	....	....	85.5	68.0	51.5
145	144	47.5	78	....	*21.0*	*100.5*	*100.5*	50.0	83.5	....	....	....	84.5	66.5	49.5
140	139	46.5	76	....	*19.0*	99.5	99.5	47.0	82.0	....	....	....	84.0	65.5	47.5
135	134	45.5	74	....	*17.5*	98.0	98.5	43.5	80.0	....	....	....	83.0	64.0	45.5
130	129	44.0	72	....	*16.0*	97.0	97.0	40.5	78.0	....	....	....	82.5	62.5	43.5
126	125	43.0	70	....	*14.5*	95.5	96.0	37.5	76.5	....	....	....	82.0	61.0	41.5
122	121	42.0	68	....	*13.0*	94.5	95.0	34.5	74.5	....	....	....	81.0	60.0	39.5
119	118	41.0	66	....	*11.5*	93.0	93.5	31.0	72.5	....	....	....	80.5	58.5	37.5
115	114	40.0	64	....	*10.0*	91.5	92.5	....	71.0	....	....	....	79.5	57.0	35.5
112	111	39.0	62	....	*8.0*	90.5	91.5	....	69.0	....	....	....	79.0	56.0	33.5
108	108	....	60	....	....	89.0	90.0	....	67.5	....	....	....	78.5	54.5	31.5
106	106	....	58	....	....	88.0	89.0	....	65.5	....	....	....	77.5	53.0	29.5
103	103	....	56	....	....	86.5	88.0	....	63.5	....	....	....	77.0	51.5	27.5
100	100	....	54	....	....	85.5	87.0	....	62.0	....	....	....	76.0	50.5	25.5
98	98	....	52	....	....	84.0	85.5	....	60.0	....	....	....	75.5	49.0	23.5
95	95	....	50	....	....	83.0	84.5	....	58.0	....	....	....	74.5	47.5	21.5
93	93	....	48	....	....	81.5	83.5	....	56.5	....	....	....	74.0	46.5	19.5
91	91	....	46	....	....	80.5	82.0	....	54.5	....	....	....	73.5	45.0	17.0
89	89	....	44	....	....	79.0	81.0	....	52.5	....	....	....	72.5	43.5	14.5
87	87	....	42	....	....	78.0	80.0	....	51.0	....	....	....	72.0	42.0	12.5
85	85	....	40	....	....	76.5	79.0	....	49.0	....	....	....	71.0	41.0	10.0
83	83	....	38	....	....	75.0	77.5	....	47.0	....	....	....	70.5	39.5	7.5
81	81	....	36	....	....	74.0	76.5	....	45.5	....	....	....	70.0	38.0	5.5
79	79	....	34	....	....	72.5	75.5	....	43.5	....	....	....	69.0	36.5	3.0
78	78	....	32	....	....	71.5	74.0	....	42.0	....	....	....	68.5	35.5	1.0
77	77	....	30	....	....	70.0	73.0	....	40.0	....	....	....	67.5	34.0	−1.5

The use of hardness scales for hardness values shown above in *italics* are not recommended by the manufacturers of hardness testing machines since they are beyond the ranges recommended for accuracy. Such values are shown for comparative purposes, only, where comparisons may be desired and the recommended machine and scale are not available.

All so-called "conversion" tables of hardness scales are based on the assumption that the metal tested is homogeneous to a depth several times as great as the depth of penetration and that the surface conditions of the pieces tested are in accordance with recommended practice.

Conversion tables dealing with hardness can be only approximate and never mathematically exact, for it must be understood that a penetration hardness test proceeds until the specimen tested supports the applied load. It is a severely cold-worked metal that actually supports the penetrator and different metals and alloys and different analyses of the same basic alloy have different work-hardening characteristics. Nevertheless, while a hardness conversion table cannot be mathematically exact, it is of considerable value to be able to compare hardness scales in a general way. It will be noted that the hardness conversion values shown for nickel and the high-nickel alloys differ slightly from those used for steels and other non-ferrous metals.

$$(48.5\ R_b \text{ to } 108\ R_b) \qquad (9)$$

$$R_b = 0.868(45T) + 31.3 \qquad (9A)$$

$$(32\ R_b \text{ to } 48.5\ R_b)$$

R_b *versus* R_g.—Range, 66 R_b to 108 R_b, 1/16-in. diameter and 1/16-in. diameter

$$R_b = 0.636\ R_g + 46.1 \qquad (10)$$

R_b *versus* R_f.—Range, 30 R_b to 108 R_b, 1/16-in. diameter *versus* 1/16-in. diameter

$$R_b = 1.741\ R_f - 97.2 \qquad (11)$$

R_b *versus* R_e.—Range, 30 R_b to 90 R_b, 1/16-in. diameter and 1/8-in. diameter

$$R_b = 1.570\ R_e - 80.0 \qquad (12)$$

R_b *versus* R_k.—Range, 30 R_b to 96 R_b, 1/16-in. diameter and 1/8-in. diameter

$$R_b = 1.098\ R_k - 13.8 \qquad (13)$$

R_c *versus* 15*N*.—Range, 1 R_c to 50 R_c, Brale and *N*-Brale

$$R_c = 2.010(15N) - 122.0 \qquad (14)$$

R_c *versus* 30*N*.—Range, 1 R_c to 50 R_c, Brale and *N*-Brale

$$R_c = 1.160(30N) - 29.0 \qquad (15)$$

R_c *versus* 45*N*.—Range, 1 R_c to 50 R_c, Brale and *N*-Brale

$$R_c = 0.875(45N) + 2.3 \qquad (16)$$

R_c *versus* R_d.—Range, −20 R_c to 50 R_c, Brale and Brale

$$R_c = 1.315\ R_d - 32.8 \qquad (17)$$

R_c *versus* R_a.—Range, −20 R_c to 50 R_c, Brale and Brale

$$R_c = 1.965\ R_a - 98.7 \qquad (18)$$

CHART

The equations, established as previously discussed, were used in calculating the chart values. This method of correlation enables one to compile by the use of these equations hardness conversions for those hardness scales covered in this paper for nickel and high-nickel alloys. Such charts or curves so compiled will be precise for all practical purposes. The Rockwell B and Rockwell C scales were chosen as a reference, since they are the most frequently used.

The "Hardness Conversion Chart for Nickel and High-Nickel Alloys" has been check tested several times by use of the equations in establishing other charts. All check work has been consistently within the range of the chart and, with few exceptions, the average has been the same value as that calculated by the equation. This applies only when tests are made on relatively homogeneous material. Deviations from the chart will be obtained when the material in test has a laminated structure; that is, either a soft core and harder surface or *vice versa*. This occurs when light gage sheets have been annealed and given a skin pass or have been cold rolled and given a skin anneal. In cases of this nature, special conversions must be developed.

It will be noted that the hardness conversion values shown for nickel and high-nickel alloys differ slightly from those used for steel and other nonferrous alloys. The conversion was developed solely for nickel and high-nickel alloys and has been established by a large representative sample covering the hardness range of these materials.

CONCLUSIONS

The relationship between a ball scale and a cone or pyramid scale has been shown to be of practical importance in this correlation. The primary factors involved and those established were that:

1. The Rockwell ball numbers for the nickel alloys are a linear function with the reciprocal of the first power of the Vickers Hardness Numerals.

2. The Rockwell "Brale" numbers are a linear function with the reciprocal of the square root of the Vickers Hardness Numerals.

3. The Rockwell ball numbers are a linear function with the reciprocal of the first power of the Brinell Hardness Numerals.

4. The Rockwell "Brale" numbers are

a linear function with the reciprocal of the square root of the Brinell Hardness Numerals.

5. All Rockwell ball scales are linear functions of one another over specified ranges.

6. All Rockwell cone scales are linear functions of one another over specified ranges.

7. The relationship between the Rockwell ball numbers and the Rockwell cone numbers can be represented as parabolic functions.

8. The relationship between the Vickers Hardness Numerals and the Brinell Hardness Numerals can be represented as a parabolic function.

9. Rockwell B *versus* Rockwell C was established as a parabolic function, valid over the range indicated, from the unmistakable significance of the relations given above as R_b *versus* Vickers, R_c *versus* Vickers, R_b *versus* Brinell, R_c *versus* Brinell, and R_b *versus* R_c relations derived from least squares studies from test data obtained from the same specimens.

10. The equations and chart represent the relations of or between the most probable values of the universes of hardness readings on the high-nickel alloys.

Acknowledgment:

The author wishes to express his appreciation to The International Nickel Co., Inc., for permission to publish the "Hardness Conversion Chart for Nickel and High-Nickel Alloys" and to the numerous individuals in the Bayonne and Huntington laboratories of The International Nickel Co. Inc., and the Wilson Mechanical Instrument Co. laboratory who did such careful work in establishing the specific and general conversions used as a part of the present study. Acknowledgment is also due the members of the Technical Service Section, Development and Research Div., The International Nickel Co. Inc.; B. B. Betty, formerly with The International Nickel Co. Inc.; S. R. Williams, Amherst College; W. T. Griffiths, Mond Nickel Co. Ltd.; V. E. Lysaght, Wilson Mechanical Instrument Co.; Harry G. Romig and George R. Gohn, Bell Telephone Laboratories; and others whose comments and suggestions regarding the original study were incorporated in this paper.

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DISCUSSION

Mr. I. V. Williams.[1]—If I understand Mr. Huston's formula for the calculation of per cent precision it is $A - B$ over the mean times 100. I think that gives us a false impression when applied to the Rockwell test, since actually on a Rockwell hardness test a high number indicates a small penetration and *vice versa.* That is, a Rockwell reading of 100 on the B scale indicates a very small penetration, but a variation of two numbers on a Rockwell reading of 100 is quite significant. On the other hand, at a Rockwell reading of zero, for which a 2 per cent tolerance would still be zero, you would have no permissable variation, while actually two numbers variation in this range is not very significant.

Mr. W. F. Hess.[2]—I should like to ask the author what comparisons he has made with other charts which are proposed to relate these various hardness scales. Are the relations established by the author much different from those already published?

Mr. F. P. Huston, Jr. (*author's closure*).—One of the reasons the per cent precision was chosen was because it was thought to compensate for the relative error in measurement. When a small unit (comparable to a large Rockwell number) is measured with a specific instrument, the relative error in measurement is greater than that in the measurement of a large unit (comparable to a small Rockwell number) when the same instrument is used. The per cent precision breaks down at zero which would affect Rockwell C. However, it works fairly well for Rockwell B above 30 Rb.

The same analysis applies to the Vickers test. A small indentation results in a large hardness numeral and *vice versa.* Thus, the percentage method provides a graded correction for the error in measurement.

The per cent precision results in too large values in some portions of specific scales; however, in the majority of cases it results in measures which past experience has shown to be about the variation between different melts of the same alloy.

The variations between this chart for nickel and the high-nickel alloys and those published by other concerns are not very large. The main deviation is one of linearity. All the relations given on the chart are linear with the exception of the Vickers: Brinell; and if the values given in the Wilson Chart 38 are plotted, it would be seen that a large number of them are curves, or non-linear relations. Since I used the linear relationship, there is a deviation from the conversion charts for brass and steel, when compared to the high-nickel alloys.

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[1] Bell Telephone Laboratories, Inc., New York, N. Y.
[2] Rensselaer Polytechnic Institute, Troy, N. Y.

SOME OF THE EFFECTS OF CADMIUM, ZINC, AND TIN PLATING ON SPRINGS*

BY JOHN R. GUSTAFSON[1]

SYNOPSIS

Very few data have been published comparing the effects of electrolytically deposited rust preventives on hardened and tempered high-carbon spring steels. Because cadmium, zinc, and tin have had the widest commercial applications, this paper deals with these three.

Fatigue tests and salt-spray tests were employed to obtain comparative data. The program, in so far as salt spray testing was concerned, was divided into two parts. The first part dealt with the springs as plated. In the second part, individual batches of springs were heated to 400, 500, 600, 700, and 800 F., then subjected to salt-spray tests.

The data indicate that cadmium is the preferable rust preventive, and could be used for a wide diversity of spring applications. Both zinc and tin are inferior to cadmium from an over-all comparison. Neither of these should be recommended for spring applications involving dynamic loading conditions.

Surface finishes have become an increasingly important subject in the application of mechanical springs. Environments such as water, steam, oil impurities, and atmospheres of a general nature which readily attack steels must often be protected against to assure maximum service from spring parts. Because of the high physical properties of spring steels when compared with non-ferrous alloys it is usually more economical to use carbon or alloy steels in the design of mechanical springs than to resort to other types of metal. Furthermore, there are some spring applications such as valve springs for marine engines which require the use of quality steels that have been protected against the elements, instead of non-ferrous alloys. The uniformity of the endurance qualities of several spring steels having been quite well established by both laboratory tests and field service prompts the selection of these materials.

Modulus characteristics coupled with high physical properties in high-carbon and alloy steels tend to yield spring parts capable of accomplishing the maxmum amount of work from the least amount of material. This weight advantage and the lesser cost per pound of steel when compared with most non-ferrous alloys offer an economic advantage, provided a surface protection can be used to overcome the deleterious effects of the attacking environments. Furthermore, steel presents fewer fabricating problems than most non-ferrous alloys, resulting in additional economic advantages.

Electrolytic plating offers an inexpensive method of applying a good rust preventive. Consequently, cadmium, zinc, and tin plates have been widely used as protective agents on spring parts. Plating adherence and hydrogen embrittlement are known variables accompanying the use of these rust preventives.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Research Department, Ford Motor Co., Dearborn, Mich.

Pre-tempered high-carbon steels are the basic materials on which our American mechanical spring industry depends. The distribution of ferric carbides in a sorbitic structure makes these steels quite susceptible to hydrogen embrittlement. Furthermore, the scale adhering to their surface presents a problem in cleaning prior to plating. Because of insufficient data on the effects of electroplating on pre-tempered spring steels, this type of material was used in this investigation.

PREPARATION OF SPRINGS

MATERIAL, DESIGN, AND FABRICATION TECHNIQUES

The springs used in this investigation were fabricated from one bundle of wire. The material was tempered valve spring wire of circular cross-section conforming to Standard Specifications for Carbon-Steel Valve Spring Quality Wire (A 230–41).[2] It had the following properties:

Size 0.162 to 0.1623 in.

Analysis, per cent:

Carbon	0.66
Manganese	0.71
Sulfur	0.025
Phosphorus	0.010
Silicon	0.23

Physical Properties:
Rockwell, 43 to 45, "C" scale.
Tensile strength, 212,000 psi. for both ends of the bundle.
Reduction of area, 62 per cent for both ends.

Metallurgical Properties:

Structure	Sorbitic and very fine-grained. (See Figs. 1(*a*) and 1(*b*)).
Inclusions	None
Decarburization:	
(1) Total	None
(2) Partial	None
Seams	None

This bundle of wire was selected from several tested because of its uniform physical properties and excellent metallurgical properties. It was fabricated into springs having the following specifications:

(*a*) 0.162-in. diameter wire.
(*b*) 1.295-in. outside diameter, ± 0.008 in.
(*c*) 5.25 total coils.
(*d*) 1.312-in. free length, ± 0.010 in.
(*e*) Closed ends and ground 1 deg. square with spring axis for 300-deg. bearing.
(*f*) Spring rate 218 lb. per inch, ± 6 lb.

The tolerances in these specifications cannot be considered commercial. They were purposely reduced to a point where individual spring selection was required. Wider dimensional tolerances would have increased the rate tolerance and in turn widened the load tolerances for the several fatigue tests. These specifications yield a theoretical stress per pound carried of 800 psi. This stress is corrected for curvature effects.

The sequence of operations used in manufacturing the springs was (*a*) coil, (*b*) strain-relieve at 725 F. for 40 min., (*c*) grind, (*d*) shot-peen, (*e*) strain-relieve at 500 F. for 40 min., (*f*) scale test. Manufacturing imperfections were kept at a minimum in order to assure uniform quality in each spring. Photomicrographs of the surface of the completed springs are shown in Figs. 2(*a*) and 2(*b*). Figure 2(*a*) shows a light coiling mark less than 0.0005 in. deep at the inside diameter. Figure 2(*b*) shows no apparent surface discontinuities at the outside diameter. Several springs were etched with a 50 per cent hydrochloric acid solution until the wire diameter was reduced to 0.156 in. A macro-examination of these etched specimens indicated that

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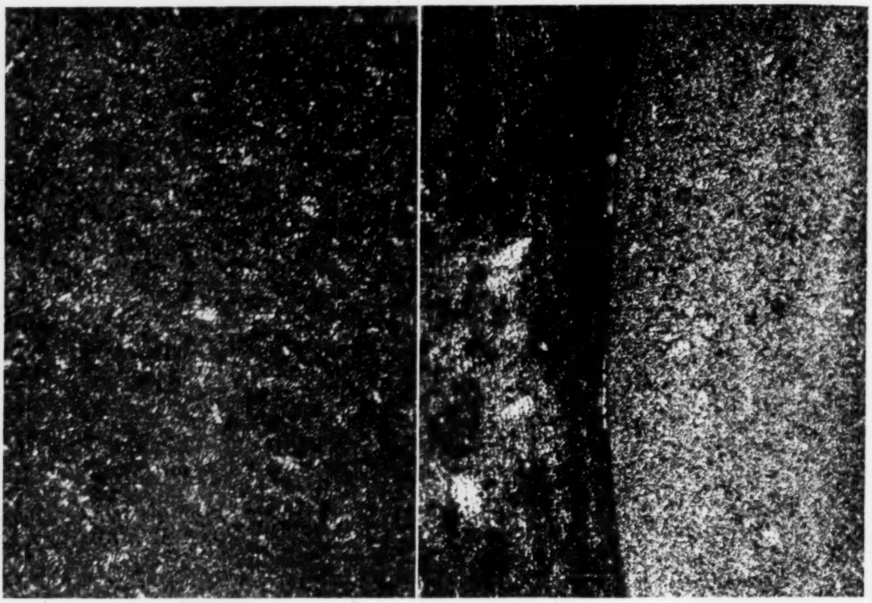
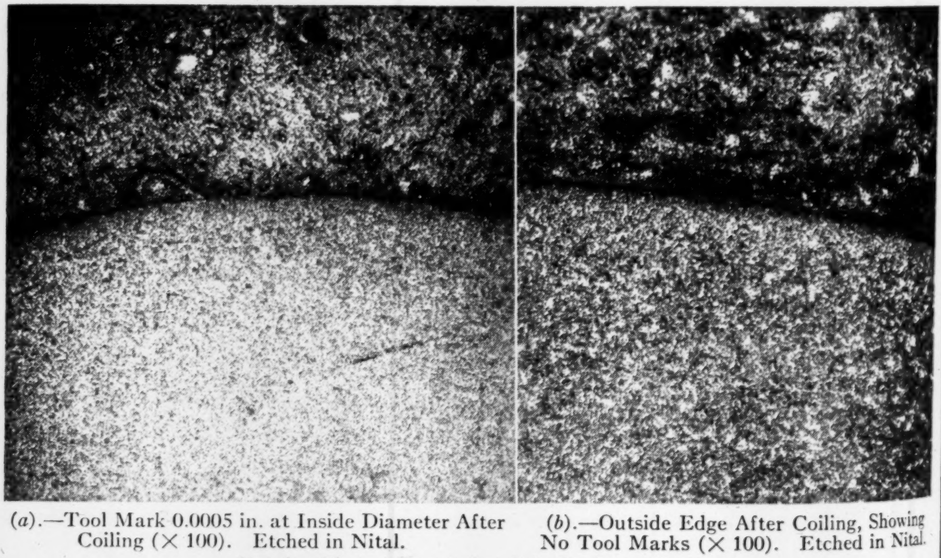
[2] 1946 Book of A.S.T.M. Standards, Part I-A, p. 202.



(*a*).—Cross-Section of Wire (× 1000). Etched in Nital.

(*b*).—Surface Condition of Wire (× 100). Etched in Nital.

FIG. 1.—Cross-Section and Surface Condition of Wire.



(*a*).—Tool Mark 0.0005 in. at Inside Diameter After Coiling (× 100). Etched in Nital.

(*b*).—Outside Edge After Coiling, Showing No Tool Marks (× 100). Etched in Nital.

FIG. 2.—Photomicrographs of the Surface of Springs.

there were no tool marks which would affect the fatigue endurance limit of the wire. Both strain-relieving operations were closely controlled. The temperature variation in each case was ± 2.5 F. and the time variation ± 1 min. The Rockwell hardness "C" scale of ten finished springs averaged 42.5 to 43.5. Rigid standards were set for the grinding operation in order to make sure the ends of the springs were not overheated. The Rockwell hardness of the ground ends was 42.5 to 43.5 "C" scale, the same as for the body of the springs. Shot-peening was done in a Pangborn Rotoblast. The shot size was 0.019 to 0.023-in. diameter, and was thrown on the springs at a rated velocity of 225 ft. per second. The time cycle for shot-peening was 40 min.

The springs were scale-tested at 1.7-in. height and to a load requirement of 51 ±1 lb. Those springs testing less than 50 lb. were thrown away and those exceeding 52 lb. were reground to meet the specified load requirement.

CLEANING AND PLATING

All springs to be plated were mechanically cleaned. This was accomplished by tumbling the springs until clean in a conventional type tumbling barrel containing a mixture of sand and sodium cyanide solution. In order to keep the springs from tangling they were strung on wires in small groups. The tumbling took approximately 1 hr. A rinse in fresh cold water to remove the cleaning solution followed, then rinsing in hot water, and drying in a centrifugal drier.

The springs were divided into three groups immediately following the cleaning operation and plated. The plating procedures were:

Cadmium Plating

(*a*) Type	barrel plating
(*b*) Chemical analysis of plating solution, oz. per gal.	
1 Cadmium	3.73
2. Sodium cyanide	14.35
3. Carbonates	3.35
4. Hydroxides	2.03
(*c*) Current density, amp. per sq. ft.	10.5
(*d*) Plating time, min.	20.0
(*e*) Roasting time, hr.	1¼
(*f*) Roasting temperature, deg. Fahr.	350
(*g*) Plating thickness, in.	0.00029 to 0.00035

Zinc Plating:

(*a*) Type	barrel plating
(*b*) Chemical analysis of plating solution, oz. per gal.	
1. Zinc cyanide	8.1
2. Sodium cyanide	6.0
3. Caustic	14.3
4. Sodium bisulphate	1.0
5. Sodium sulphide	0.065
(*c*) Current density, amp. per sq. ft.	15
(*d*) Plating time, min.	30
(*e*) Roasting time, hr.	2
(*f*) Roasting temperature, deg. Fahr.	350
(*g*) Plating thickness, in.	0.0003 to 0.00034

Tin Plating:

(*a*) Type	barrel plating
(*b*) Chemical analysis of plating solution, oz. per gal.	
1. Sodium stannate	17.9
2. Sodium hydroxide	2.0
3. Sodium acetate	3.06
4. Sodium peroxide (100 val.)	0.06
(*c*) Current density, amp. per sq. ft.	17.0
(*d*) Solution temperature, deg. Fahr.	160
(*e*) Plating time, min	45
(*f*) Roasting time, hr.	2
(*g*) Roasting temperature, deg. Fahr.	350
(*h*) Plating thickness, in.	0.0003 to 0.00035

The plating thicknesses for cadmium and zinc were determined by the chromic acid drop test method. The thickness of the tin plate was obtained by mounting cross-sections of plated springs in bakelite, polishing, and inspecting microscopically, using a micrometer eyepiece. The roasting time was lengthened for the zinc and tin plated springs in order to try to overcome the effects of increased hydrogenation taking place in these types of platings.

Every care was taken to obtain uniform plating. The baths were filtered and the electrical equipment checked before plating each batch of springs. The barrel-plating method was employed because it is more economical than still-plating if the parts to be plated lend themselves to this method. These springs plated quite satisfactorily. Good metal adherence was obtained due in great part to the method of cleaning employed. Cadmium is very susceptible to blistering if the surface to be plated is not thoroughly clean, whereas zinc and tin seem to be slightly less affected by small surface dirt particles than cadmium, and consequently are easier to plate. This may be due to the fact that their baths tend to act as additional cleaning agents. Roasting will exaggerate a blistered condition if one exists. In every case the plating was free from blisters and had good adherence.

A comparison of the plating procedures brings out the fact that the current density and time for an equal plating thickness is the least for cadmium. Because the tin plating solution must be heated, tin plating is the most expensive.

Zinc, cadmium, and tin are electronegative elements, decreasing in this characteristic in the order named. Both zinc and cadmium have the same lattice structure, being hexagonal close packed, whereas tin is body-centered tetragonal. The melting point of zinc is highest and that of tin lowest. There are many similarities and contrasts in comparing the physical and chemical qualities of these three elements. Likewise they should yield similarities and contrasts in any study of their effects on a base metal when electrolytically deposited.

FATIGUE ENDURANCE TESTS

Fatigue tests were carried out on the plated springs in order to compare the effects of the electrolytically deposited metals on *S-N* relationships, the degree of scatter, and the fatigue endurance limit. The testing machine had been especially designed for the type of information required, and to accommodate the springs fabricated for testing purposes. A schematic drawing of this machine is presented in Fig. 3. It is equipped to test 32 springs, eight rows of

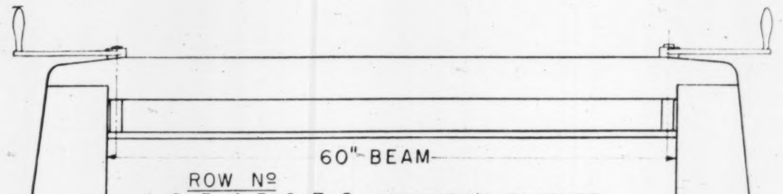


FIG. 3.—Diagram of Testing Machine.

four springs each, thereby testing four springs at each stress range. The design of this testing unit is such that at the beginning of each test the initial stress for every row is the same, and the final stress at the end of the stroke varies directly from each row to the pivoting point *D* of the actuating ram. The cam shaft is circular, being offset to yield a maximum stroke of 1.0 in.

TEST METHODS

Preliminary tests using unplated springs were carried out before making fatigue tests on specimens prepared for this investigation. These preliminary tests established the maximum and minimum stresses employed to obtain data on the plated springs.

TABLE I.—SPRING DATA BEFORE FATIGUE TESTING.

Row	Minimum Loaded Height, in.	Maximum Loaded Height, in.	Before Testing			
			Minimum Average Load, lb.	Maximum Average Load, lb.	Minimum Average Stress, psi.	Maximum Average Stress, psi.
No. 1	1.856	1.100	18	180	14 400	144 000
No 2	1.856	1.149	18	171	14 400	137 000
No. 3	1.856	1.198	18	162	14 400	129 600
No. 4	1.856	1.247	18	152	14 400	121 600
No. 5	1.856	1.296	18	141	14 400	111 800
No. 6	1.856	1.345	18	131	14 400	104 800
No. 7	1.856	1.394	18	119	14 400	95 200
No. 8	1.856	1.443	18	108	14 400	86 500

There were 200 unplated springs set aside for testing purposes. Consequently four different groups were used to obtain fatigue data. Every spring in each of the groups was magnafluxed and inspected at 7 magnifications for seams, pits, tool marks, or any other surface imperfections that might cause premature failure. All springs were then suitably identified. This identification classified the springs according to row, that is, stress range, and the number in the row for the eight different stress ranges. Each spring was load-tested at its two loaded heights and this information recorded. Because of previous selection by scale testing, all springs tested to loads within ±1 lb. for all eight stress ranges. Finally, the springs were load-cycled at the test heights determined from the preliminary tests. (See Table I for loaded heights, loads, etc.)

The fatigue tests were ended at 10,000,000 cycles. When individual specimens broke, the number of cycles to failure was recorded. All tests were interrupted at 150,000 and 5,000,000 cycles and unbroken springs scale-tested. All *S-N* data are based on the average load of the final load test before failure, or the average loads of the individual rows after 10,000,000 cycles. Therefore the endurance limit curves take into account the average permanent set of each spring row since there were a sufficient number of unbroken springs in each row after 150,000 cycles to arrive at an average load and consequently an average stress. Results are based on test data obtained from eight springs cycled at the eight stress ranges.

TABLE II.—PLAIN FINISH SPRING.
(Average load per row)

Row	Capacity at Initial and Final Test Heights, lb.						Unbroken Springs
	150,000 Cycles		5,000,000 Cycles		10,000,000 Cycles		
	Minimum	Maximum	Minimum	Maximum	Minimum	Maximum	
No. 1	2	168	....	....	....	....	0
No. 2	4	161	....	....	....	....	0
No. 3	7	154	5	150	....	....	0
No. 4	12	148	9	145	7.5	143	4
No. 5	14	138	12	136	10	134	7
No. 6	16	129	14	127	14	127	8
No. 7	16	118	16	117	16	117	8
No. 8	17	107	17	107	17	107	8

RESULTS OF FATIGUE TESTS

The testing heights and loads at the testing heights are given in Table I. The average load was held to ±1 lb. in both the minimum and maximum loaded posi-

TABLE III.—CADMIUM PLATED SPRINGS.
(Average load per row)

Row	Capacity at Initial and Final Test Heights, lb.						Unbroken Springs
	150,000 Cycles		5,000,000 Cycles		10,000,000 Cycles		
	Minimum	Maximum	Minimum	Maximum	Minimum	Maximum	
No. 1	1	164	....	....	....	....	0
No. 2	4	159	1.5	153.5	....	....	0
No. 3	8	154	5	150	4	148	1
No. 4	11	148	9	144	9	144	6
No. 5	15	139	12	136	12	136	7
No. 6	16	129	15	127	15	127	8
No. 7	17	118	16	117.5	16	117.5	8
No. 8	18	108	17	107	17	107	8

TABLE IV.—ZINC PLATED SPRINGS.
(Average load per row)

Row	Capacity at Initial and Final Test Heights, lb.						Unbroken Springs
	150,000 Cycles		5,000,000 Cycles		10,000,000 Cycles		
	Minimum	Maximum	Minimum	Maximum	Minimum	Maximum	
No. 1	1	162	....	....	....	....	0
No. 2	4	158	....	....	....	....	0
No. 3	9	154	5	150	4	148	1
No. 4	11	145	8	141	7.5	139.5	1
No. 5	13	136.5	....	....	....	....	0
No. 6	15	127.5	13.5	126	12	124	5
No. 7	16	116	15	115	15	115	5
No. 8	16.5	106	16	105	16	106	6

TABLE V.—TIN-PLATED SPRINGS.
(Average load per row)

Row	Capacity at Initial and Final Test Heights, lb.						Unbroken Springs
	150,000 Cycles		5,000,000 Cycles		10,000,000 Cycles		
	Minimum	Maximum	Minimum	Maximum	Minimum	Maximum	
No. 1	1	163	....	....	....	....	0
No. 2	3.5	157	....	....	....	....	0
No. 3	7	155	....	....	....	....	0
No. 4	11	147	8	144	6	142	3
No. 5	13	136	11	134	10	133	5
No. 6	16	129	14	127	14	127	6
No. 7	17	118	16	117	16	117	7
No. 8	17	106	16	106	16	106	7

tion. The initial and final loaded heights represent the heights at which each row was tested for endurance characteristics, and they were maintained throughout the several tests. The load was allowed to vary as the springs took permanent set. The average load for each row after 150,000, 5,000,000 and 10,000,000 cycles is given in Tables II, III, IV, and V.

The maximum stress, minimum stress and stress range for each type of finish, and for each row are given in Table VI. This information is based on average load conditions before breakage of every spring in each row, and is computed from the information in Tables II to V, inclusive.

The stress ranges of Table VI are combined with the number of cycles to failure for each spring in each row to arrive at the *S-N* curves and the degree of scatter presented in Figs. 4, 5, 6, and 7.

A comparison of the lower limit curves of Figs. 4 to 7, inclusive, is given in Fig. 8. These are pertinent engineering data be-

TABLE VI.—TEST STRESSES IN POUNDS PER SQUARE INCH.

Row	Plain			Type of Finish								
				Cadmium			Zinc			Tin		
	Maximum Stress, psi.	Minimum Stress, psi.	Stress Range, psi.	Maximum Stress, psi.	Minimum Stress, psi.	Stress Range, psi.	Maximum Stress, psi.	Minimum Stress, psi.	Stress Range, psi.	Maximum Stress, psi.	Minimum Stress, psi.	Stress Range, psi.
No. 1	135 000	1 600	133 000	131 000	1 000	130 000	130 000	1 000	129 000	131 000	1 200	130 000
No. 2	129 000	3 200	126 000	123 000	1 200	122 000	127 000	3 000	124 000	126 000	3 000	123 000
No. 3	120 000	4 000	116 000	118 000	3 200	115 000	119 000	5 000	114 000	120 000	4 000	116 000
No. 4	114 000	6 000	108 000	115 000	7 000	108 000	111 000	6 000	105 000	113 000	6 000	107 000
No. 5	107 000	8 000	99 000	109 000	9 600	99 000	105 000	8 000	97 000	107 000	8 000	99 000
No. 6	101 000	11 200	90 000	102 000	12 000	90 000	99 000	10 000	89 000	100 000	11 000	89 000
No. 7	94 000	12 800	81 000	94 000	13 000	81 000	93 000	12 000	81 000	94 000	12 500	81 000
No. 8	86 000	13 600	72 000	86 000	13 600	72 000	85 000	13 000	72 000	86 000	13 500	72 000

cause they show graphically the difference in *S-N* relationships produced by the effects of plating. This is the information which should be applied to guide one's thinking in arriving at a suitable stress range for one of the plated rust preventives in terms of life expectancy of the spring. For example, assume a life expectancy of 300,000 cycles from a coil spring and that the environment is such that cadmium, tin, or zinc can be used as the rust preventive. The stress ranges for tin, zinc, and cadmium are 62,000, 64,000, and 116,000 psi., respectively. These coupled with the maximum stresses given in Table VI establish the maximum and minimum stress to which a coil spring can be designed, and meet the life requirements expected. In using Table VI as a guide, the final stress for a cadmium-plated spring could be 120,000 psi. and the initial stress 4000 psi. For

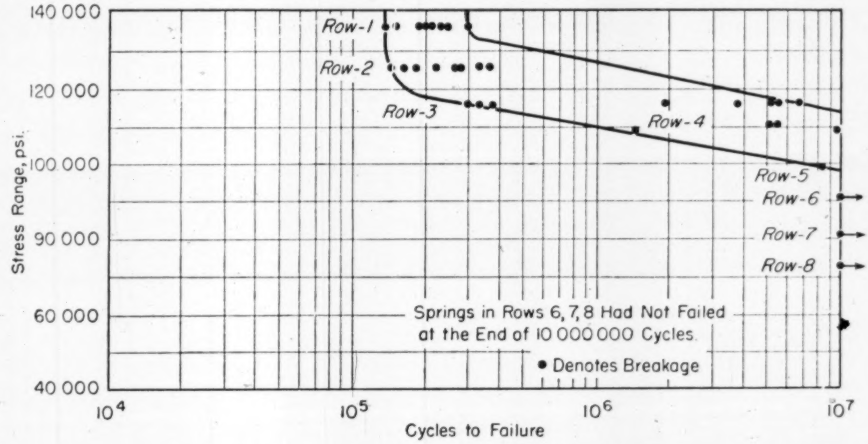


FIG. 4.—*S-N* Curve and Scatter Diagram for Plain Finish.
A.S.T.M. A 230–41 steel.

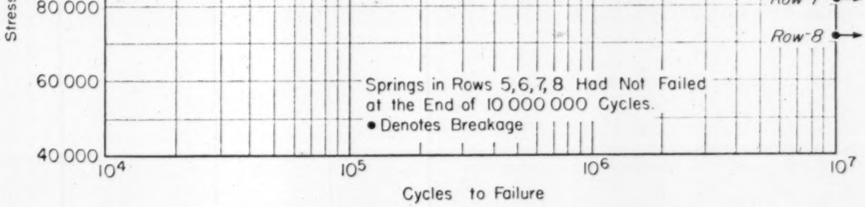


FIG. 5.—*S-N* Curve and Scatter Diagram for Cadmium-Plated Material.
A.S.T.M. A 230–41 steel.

either zinc or tin the maximum permissible stress would have to be less than 85,000 psi., whereas the initial stress would have to be greater than 13,000 psi. In order to do the same amount of work it takes more material with a zinc or tin finish for the same life expectancy, thereby increasing the unit cost of each spring.

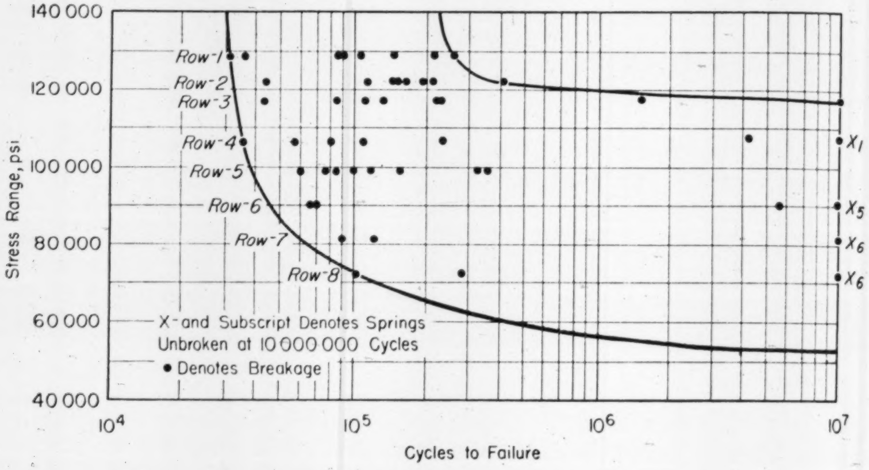


FIG. 6.—*S-N* Curve and Scatter Diagram for Zinc-Plated Material.
A.S.T.M. A 230-41 steel.

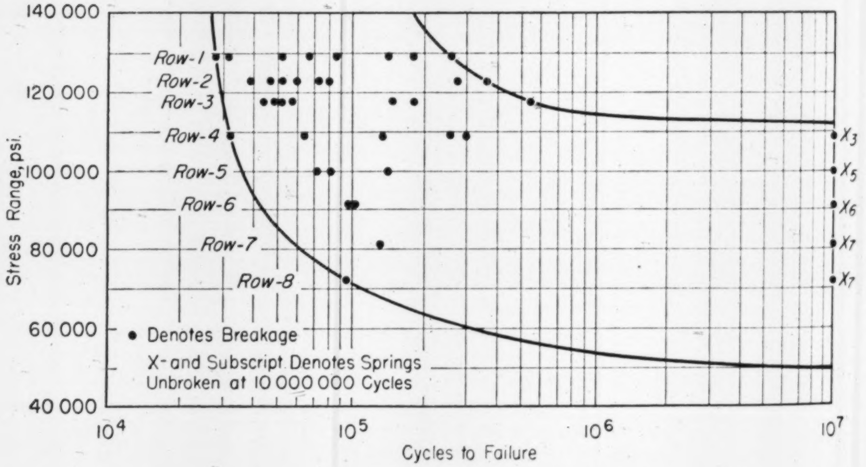


FIG. 7.—*S-N* Curve and Scatter Diagram for Tin-Plated Material.
A.S.T.M. A 230-41 steel.

Table VII gives the average percentage load loss for each row and for each finish. This information is based upon the percentage differences between the load data in Table I and Tables II, III IV, and V.

A comparison of the data compiled on plain finished springs agrees with data obtained by other investigators on tempered valve spring wire. Therefore it can be assumed that the material selected was of excellent quality and uniform in all its metallurgical and physical aspects. The endurance limit of 107,600 psi. final stress and 8000 psi. initial stress at the end of 10,000,000 cycles satisfies limits found from the Goodman diagram for shot-peened coil springs fabricated to a valve-spring quality. This indicates that both workmanship and the cold-working from shot-peening were satisfactory.

TABLE VII.—PERCENTAGE OF LOAD LOSS PER ROW.

Row	Plain Finish	Cadmium-Plated	Zinc-Plated	Tin-Plated
No. 1........	7.0	8.6	7.9	8.0
No. 2........	6.5	8.2	9.3	6.9
No. 3........	6.5	6.3	6.0	4.0
No. 4........	6.0	5.6	5.5	5.0
No. 5........	4.5	3.5	1.9	3.0
No. 6........	2.8	1.9	4.0	2.0
No. 7........	1.6	1.4	2.5	1.5
No. 8........	0.46	1.3	1.0	0.63

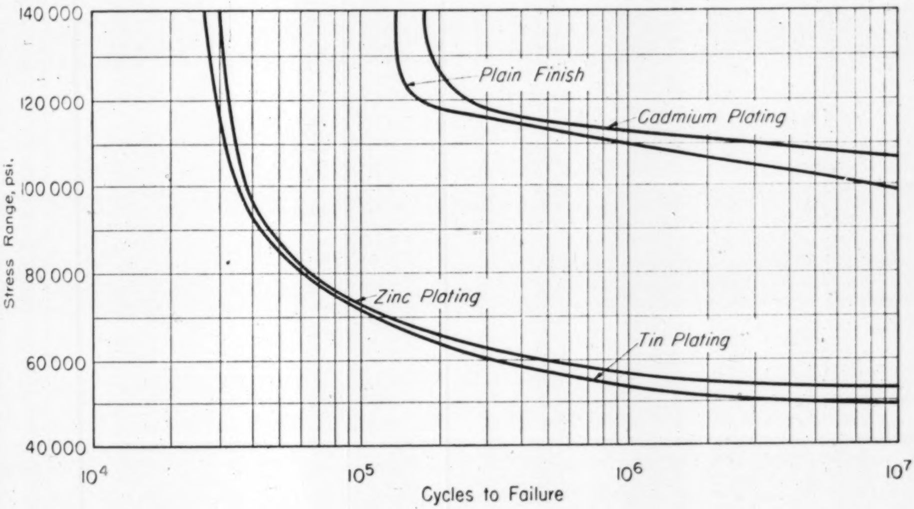


FIG. 8.—A Comparison of Lower Limit Curves.
A.S.T.M. A 230-41 steel.

Cleaning prior to plating has offered a challenge to the spring industry for many years. Chemical cleaners, whether basic or acid, have either cleaned insufficiently or attacked the base metal, causing hydrogen embrittlement or produced chemical reactions which in turn have markedly reduced spring life. The author has investigated many types of cleaning methods, and the mechanical method used in this investigation is actually beneficial in increasing the fatigue-endurance limit of tempered spring steel. Wet sand tumbling cold-works and polishes the surface, thereby reducing stress concentrations. A comparison of fatigue-test results between plain springs and cadmium-plated springs gives evidence bringing out the beneficial effects of the cleaning procedure (see Figs. 4 and 5). The plain springs were not cleaned after shot-peening.

The various platings used were of excellent quality. All finishes were free from blisters and had good adherence.

Additional *S-N* curves are necessary to complete the information regarding endurance limits at increased initial stresses and finite life expectancy at stress ranges within the limits of finite life. Such a comprehensive investigation could not be carried out without first exploring some of the effects of cadmium, zinc, and tin finishes on coil springs.

CONCLUSIONS

Plain Finish (Fig. 4):

The nature of the curves and the degree of scatter indicate that normal results were obtained. A comparison of Fig. 4 with the number of unbroken springs at the end of 10,000,000 cycles, given in column 8, Table II shows that the data yield a progressive fracture pattern. The information establishes a definite fatigue endurance limit.

Cadmium-Plated Finish (Fig. 5):

(*a*) Cadmium plating as carried out for this investigation had no effect on the fatigue life of the springs.

(*b*) The data of Fig. 5 and Table III, column 8, yield a progressive pattern establishing a fatigue endurance limit.

(*c*) Cadmium plating is recommended as a rust preventive on springs loaded dynamically if cadmium is suitable for the environment.

Zinc-Plated Finish (Fig. 6):

(*a*) Zinc plating as carried out for this investigation had a marked effect on the fatigue life of the springs. The degree of scatter is so wide that it would be impossible to arrive at a probability factor to determine life in terms of a stress range. The final column in Table IV brings out the fact that the tendency for breakage is not progressive as the stress range increases.

(*b*) An examination of the fractures definitely established the cause of failure to be hydrogen embrittlement.

(*c*) Zinc plating should not be recommended as a rust preventive for springs subjected to dynamic loading conditions.

Tin-Plated Finish (Fig. 7):

Those conclusions drawn for zinc plating also hold true for tin Although the final column in Table V indicates progressive failure as the stress range increases, a comparison of these data with that on the *S-N* curve, Fig. 7, brings out the fact that breakage in terms of stress range does not follow a progressive pattern. Breakage occurred at low stress ranges before it occurred at higher stress ranges. Therefore it can be concluded that the surface stress concentration produced by tin plating were not uniform in character.

TABLE VIII.—FATIGUE-ENDURANCE RANGES.

Type of Finish	Initial Stress	Final Stress	Stress Range
Plain	8 000	107 600	99 600
Cadmium.........	9 200	108 800	99 600
Zinc..............	None	None	None
Tin...............	None	None	None

Elastic Limit:

The data of Table VI which compare the effects of the various finishes on permanent set indicate that the elastic limit of the base metal was not affected by the electrolytic finishes.

SALT-SPRAY TESTS

One generally used method of conducting an accelerated test on rust preventives is to use salt spray. Although it is argued that cadmium chloride is less soluble than zinc chloride, thereby favoring cadmium, the author feels that other factors are more important in arriving at conclusions based on a comparative salt-spray test. Since cadmium plating tends to be more porous and more susceptible to blistering unless properly deposited, it may yield unfavorable results in any competitive environment, whether this be salt spray or in industrial, urban or rural exposures. Therefore a perfectly clean surface, prior to plating, and one which will yield good adherence is most important. Another factor to be considered is the final treatment of the exposed surface of the rust preventive.

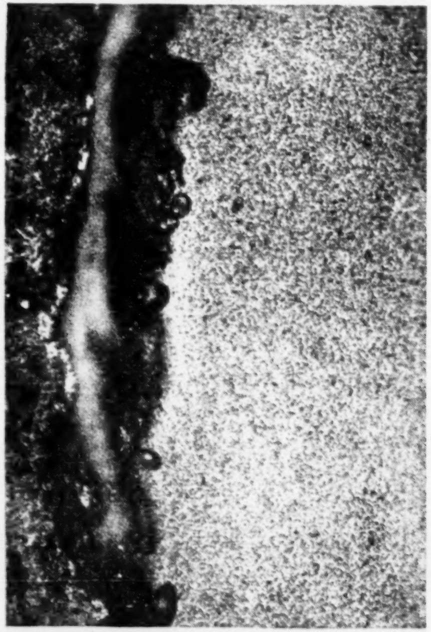


FIG. 9.—Cadmium Plating (× 100). Etched in Nital. Pit depth, 0.004 to 0.006 in. at end of 1000 hr. in salt spray.

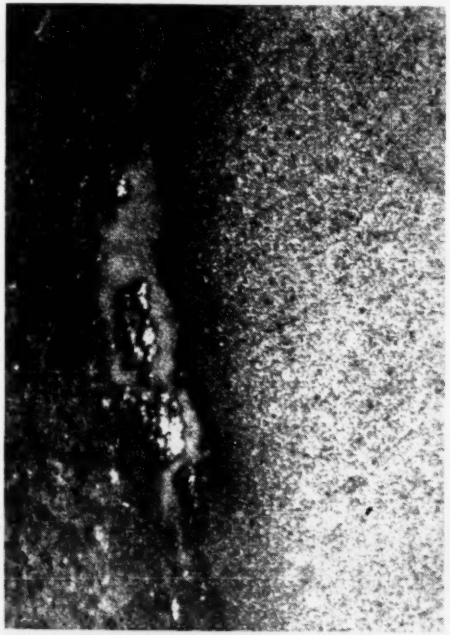


FIG. 10.—Zinc Plating (× 100). Etched in Nital. Pit depth, 0.004 to 0.006 in. at end of 1000 hr. in salt spray.

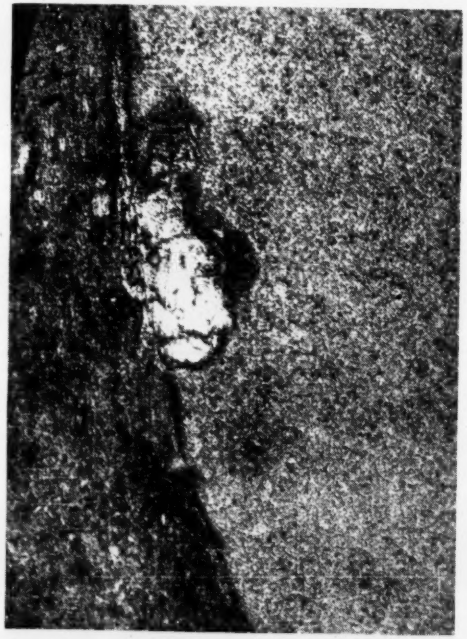


FIG. 11.—Tin Plating (× 100). Etched in Nital. Pit depth, 0.004 to 0.010 in. at end of 1000 hr. in salt spray.

For this comparison (see operation sequences, given in "Cleaning and Plating" section), all plated springs were roasted at 350 F. This temperature was below that necessary to form surface oxides which in turn would have decreased the plated thickness and increased the rate of corrosion. A final consideration and an important one is the metallurgical structure and physical properties of the base metal. Since a tempered high-carbon steel was used for this investigation, interpretation of data and conclusions drawn apply to this grade of material.

Not only is the rate of rusting faster for a pre-tempered high-carbon steel when compared with either cold- or hot-rolled low-carbon steels, but the tendency toward pitting is also accelerated once the protective coating has been destroyed. This factor must also be considered in evaluating the merits of a rust preventive.

Both zinc and tin are more easily deposited than cadmium, and adhesion and blistering are not too difficult to avoid. However, each must be electrodeposited within that current density range which will not show evidence of too rapid deposition of the electrolyte.

Equipment:

The equipment consisted of an asphalt-lined wooden salt-spray cabinet, having a built-in salt solution tank. The air to the nozzle spray passed through a filter which removed all moisture and other contaminations. The nozzle pressure was held at 15 psi. and the cabinet temperature maintained at 95 ± 5 F. Distilled water and Morton's granulated salt were used to make up the salt solution and was mixed to a specific gravity of 1.15. The salt solution tank inside the cabinet was filled every 24 hr.

Test Methods:

Ten springs of each finish were used for the salt spray tests. These springs were placed on glass rods in such a manner that they did not touch one another or the sides of the salt-spray cabinet. At the end of each 100 hr. the springs were removed from the cabinet, thoroughly washed in distilled water, and macro-examined at a magnification of seven to determine the percentage of the surface that had corroded. Although each spring was individually examined, the percentage values arrived at were collective in that they were based on the average of the ten springs of each type of plating. It was felt that a more uniform comparison could be obtained from an average of ten springs than to compare the worst and best condition of springs from each group. All springs were exposed to salt spray under the conditions outlined for 1000 hr.

Upon completion of the salt-spray test, a cross-section from one spring of each finish was micro-inspected. The depth of pits was obtained from that part of the spring which appeared to represent an average corroded condition. Representative cross-sections are shown in Figs. 9 to 11, inclusive. The remaining nine springs of each finish were etched in a 50 per cent hydrochloric acid solution and macro-examined at a magnification of seven.

Salt-Spray Test Results:

The percentage of corroded area at the end of each 100 hr. is shown graphically in Fig. 12. It is interesting to note that the cadmium-plated springs did not start corroding until after 500-hr. exposure to salt spray. This is an important consideration, because the springs had been thoroughly washed in distilled water four times, thereby tending to remove some of the protective compounds credited to extending the life of cadmium as a rust preventive. The zinc- and tin-plated springs, having been treated in a like manner, had respectively 15 and 35 per cent of their surfaces corroded after 500-hr. exposure. Between 500 and 800 hr. salt-spray exposure, zinc had almost completely broken down with a 75 per cent corroded area *versus* 30 per cent for cadmium. Tin plating broke down more quickly than the zinc plate, having rusted slightly at the end of the first 100 hr.

It is admitted that a probability of error exists in estimating the percentage of corroded surface. The method of averaging the effects of salt-spray exposure on ten springs, however, seems to have held this error to a minimum. A smooth curve could be plotted through the several averages obtained from this method. The averages for the tin finish at the end of 300 and 700 hr. show the greatest variation.

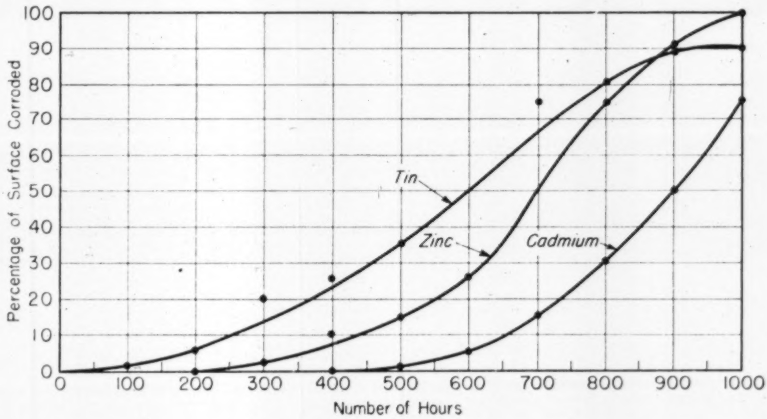


FIG. 12.—Resistance to Salt Spray.

A macro-examination of the etched springs after 1000-hr. exposure to salt spray yielded the following results:

1. Cadmium............	75 per cent of surface covered with light pits.
2. Zinc.................	Surface honeycombed with light rust pits. 100 per cent of surface covered with light pits.
3. Tin..................	Surface completely covered with rust pits. Twenty-five per cent of surface had pitted to a minimum depth of 0.010 in.

Conclusions:

Based on the data, it can be concluded that cadmium offered the greatest resistance to corrosion. Zinc was a favorable second but is likely to break down more rapidly than cadmium once corrosion starts. This is an important comparison because it indicates that spring replacement would be greater for zinc than for cadmium-plated springs for like environments. The evidence obtained supports this statement. Assuming that 50 per cent breakdown in the rust preventive corresponds to spring failure, zinc lasted 700 hr. whereas cadmium lasted 900 hr. Tin was the least favorable from a standpoint of resistance to salt spray. However, there are special environments, some of an organic nature, for which tin is peculiarly suited. Tin-plated springs, when used under these conditions, will present a replacement problem which should be considered if it must be adopted as a rust preventive.

Comments:

Corrosion fatigue is important in the application of any spring and is a subject of considerable controversy. Supplementing data already presented, the author exposed several cadmium-plated and zinc-plated springs to the out of doors for one year. The general atmosphere was that found in an average urban community being contaminated with smoke, factory residues, etc. At the end of one year the cadmium-plated springs had 12 per cent of the area corroded and zinc-plated springs 29 per cent. This is equivalent to salt-spray findings at the end of approximately 650 hr. This finding is not in complete agreement with published information on the subject and may be due in great part to the methods used in cleaning and plating the springs as well as the baking operation after plating. It might also be argued that barrel plating being somewhat slower than still plating results in a tougher surface which in turn is more resistant to the elements, and in the case of cadmium, increased toughness was sufficient to reverse the findings of other investigators.

SALT-SPRAY TESTS OF SPRINGS SUBJECTED TO ELEVATED TEMPERATURES

The effects of elevated temperatures on rust preventives is not an unusual problem in the proper functioning of coil springs. Very few investigations have been carried out, however, to determine the effects of elevated temperatures on cadmium, zinc, and tin platings and to draw comparisons of their ability to resist corrosion or to determine at what temperatures they lose their effectiveness.

It is known that these elements react chemically in different ways at elevated temperatures, and these reactions should influence the results of a temperature study on their ability to resist atmospheric attacks. For example, the melting point of tin is 449 F., and when it is heated above this temperature in air a stannic oxide is formed. This oxide changes to a gas and is driven off as the temperature rises. Cadmium has a melting point of 608 F., whereas zinc melts at 788 F. These metals differ from tin in that they form oxides when heated in air before their melting points are reached. Traces of these oxides are produced at 400 F., and their formations are accelerated at 500 F. As the temperature rises above 500 F., zinc oxide tends to change from a solid to a gas, whereas this condition does not appear in cadmium until the temperature exceeds 608 F. Because of these temperature effects it was decided to compare the salt-spray resistance of cadmium-, zinc-, and tin-plated springs that had been subjected for 4 hr. to 400, 500, 600, 700, and 800 F. Preliminary tests indicated that 4 hr. was sufficiently long to form the oxides of the three elements.

Equipment:

The same equipment was used in this investigation as was used to obtain a comparison of salt-spray on coil springs as plated.

Test Methods:

Fifty springs of each type of plating were used in this investigation. Each group of fifty springs was divided into five subgroups of ten springs each. One subgroup of each finish was heated in a recirculating air furnace at 400 F. for 4 hr. A second subgroup of each finish was then heated at 500 F. for 4 hr. and so on until the fifth subgroup of each finish had been heated at 800 F. for 4 hr. The springs comprising the fifteen groups were strung on glass rods and placed in the salt-spray cabinet in such a manner that they neither touched one another or the side of the cabinet. At the end of 75 hr. the springs were removed from the cabinet, washed in distilled water, macroexamined at a magnification of seven, and the results, based on an average of the ten springs in each subgroup, were

TABLE IX.—RUST AREA IN PER CENT AND RUST PITTED CONDITION.

Temperature, deg. Fahr.	Cadmium Plate.	Zinc Plate	Tin Plate
		75 HR.	
400	None	None	2 per cent light pits
500	None	None	20 per cent medium pits
600	None	None	20 per cent medium pits
700	None	None	20 per cent medium pits
800	None	2 per cent light pits	20 per cent medium pits
		150 HR.	
400	None	None	5 per cent light pits
500	None	None	35 per cent medium pits
600	None	2 per cent light pits	35 per cent medium pits
700	None	2 per cent light pits	35 per cent medium pits
800	None	5 per cent light pits	50 per cent deep pits
		225 HR.	
400	None	2 per cent light pits	10 per cent medium pits
500	None	2 per cent light pits	50 per cent deep pits
600	None	5 per cent light pits	50 per cent deep pits
700	None	5 per cent light pits	50 per cent deep pits
800	5 per cent light pits	10 per cent medium pits	
		300 HR.	
400	2 per cent None	10 per cent medium pits	25 per cent deep pits
500	2 per cent None	10 per cent medium pits	
600	5 per cent light pits	20 per cent deep pits	
700	5 per cent light pits	20 per cent deep pits	
800	10 per cent medium pits	25 per cent deep pits	

recorded. This procedure was repeated at the end of every 75 hr. All tests were to be concluded at the end of 300 hr. or at 50 per cent average corroded area of any of the subgroups. Tin-plated springs heated at 500, 600, and 700 F. were removed at the end of 225 hr., each subgroup having corroded 50 per cent. Tin-plated springs heated at 800 F. were removed at 150 hr. since the springs had 50 per cent of their areas corroded.

Salt-Spray Test Results:

Results at the end of each 75-hr. period are outlined in Table IX. The rested area is given in percentage of total area, and the severity of the rusted condition is classified as light pits, medium pits, and deep pits. Light pits define a condition whereby the salt-spray attack was less than 0.0015 in. deep whereas medium pits are less than 0.004 in. deep and deep pits exceed 0.008 in. The data are grouped in such a manner that a comparison can be made of the effects of the elevated temperatures on the three rust preventives.

A graphic presentation of the percentage of corroded area *versus* time of exposure is presented in Fig. 13, and covers each of the five temperatures examined. The rate of corrosion is aptly brought out by the graphs, and this information coupled with the data in Table IX offers a means of diagnosing some of the effects

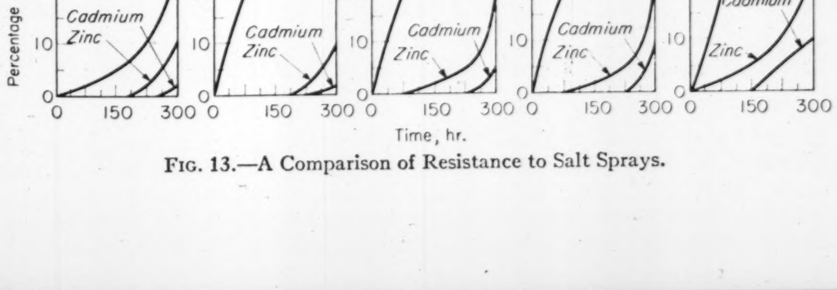


FIG. 13.—A Comparison of Resistance to Salt Sprays.

of elevated temperatures on the ability of cadmium, zinc, and tin plating to resist corrosion.

The chart, Fig. 14, shows the percentage of corroded area at the end of 300 hr. or 50 per cent deterioration due to rusting. It combines results obtained on springs as plated with those at elevated temperatures.

Conclusions:

The data bring out the fact that cadmium was superior to either zinc or tin for all the various temperatures employed in this investigation. It could be used with reasonable assurance as a rust preventive at temperatures to 600 F. Zinc, on the other hand, shows evidence of breaking down at 400 F. and to oxidize readily above that temperature. Therefore, zinc should not be used to resist corrosion at temperatures exceeding 400 F. Tin plating produced unfavorable results at every temperature and broke down completely on all springs subjected to temperatures exceeding 400 F.

Figure 14 offers a comparison of the effects of temperatures on each of the three finishes. A significant finding brought out by these charts is that the baking temperature, after electroplating cadmium, zinc, or tin, should be less than 400 F. for maximum rust-prevention characteristics.

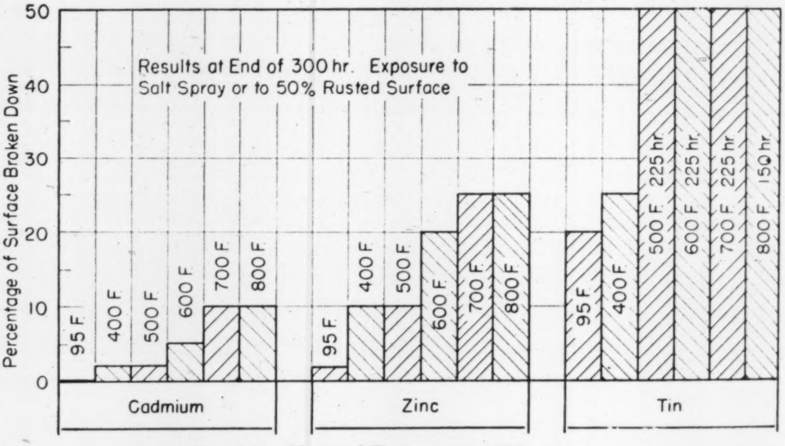


FIG. 14.—Chart of Temperature Effects.

SUMMARY

Corrosion costs the consumer millions of dollars every year, and this cost can partially be overcome by the wise selection of rust preventives. There are many applications of metals where failure is due to corrosion fatigue, and where the metal could have given many years of additional service had the selection of the proper rust preventive been made. Whenever the life of an article can be lengthened, and consequently its worth and usability enhanced by increased longevity, it is usually more economical to apply a rust proofing than to count on replacing the item. Replacement is always more expensive than prevention.

Considerable progress has been made in finding ways to retard rusting. Continued research in this field is necessary to classify harmful atmospheres and correlate them with suitable protective coatings.

DISCUSSION

MR. GUSTAF SODERBERG.[1]—I have one comment and one question. The comment is with regard to the oxidation of the cadmium. The temperature at which oxidation begins depends considerably on the purity of the cadmium. The presence of small amounts of nickel, for example, in the cadmium—which is quite common—will raise the temperature at which oxidation begins.

The question is—was any work ever done on lead plating of springs? During the war, of course, cadmium was scarce, and so was tin, and we tried to do as much as we could with lead.

MR. S. B. ASHKINAZY.[2]—I should like to ask Mr. Gustafson whether he has done any work in regard to seeing what the effect on fatigue life would be of plated springs that had been in service for some time.

As I see it, all fatigue endurance tests were conducted immediately after plating and prior to the subjecting of the springs to the salt-spray tests.

Although we note from Mr. Gustafson's work that cadmium plate has no effect on fatigue endurance properties on freshly-plated springs, and zinc and tin do, we do not know what effect the cadmium plate has on fatigue endurance properties after it has been in service for some time and has had a chance to powder and produce an unsmooth surface which we know considerably affects fatigue life.

How would cadmium-plated springs compare with nonplated springs under those conditions?

CHAIRMAN SAM TOUR.[3]—This paper compares the same thicknesses of cadmium, zinc, and tin throughout. It is doubted that this is in conformity with commercial practice in cadmium, zinc, and tin plating. Normally, much less cadmium-plate thickness is used than zinc-plate thickness.

This paper arrives at conclusions which, let us say, are complimentary to cadmium, and uncomplimentary to zinc and tin with the apparent conclusion that the results are incontrovertible. No investigation was made of the different methods of plating each of these metals to determine whether or not zinc and tin could be plated at different current densities and at different temperatures, etc., to avoid the deleterious effect which seems to have been found under the conditions used for the work reported in this paper. In other words, it would seem to me that this paper is only a report of the results obtained under the conditions under which the plating was carried out, and should not be considered as having proved the conclusion that, under all conditions, cadmium plating is superior to either zinc or tin for protection of springs.

MR. R. C. A. THURSTON[4] (*by letter*).—Mr. Gustafson is to be congratulated on an excellent piece of work in a relatively unexplored field. While the effect of numerous platings on the fatigue strength of different steels has been studied by many research workers, there is little technical literature concerning the practically important problem of small helical springs. The author's investigation was thorough and sufficiently comprehensive to justify his conclusions, the principal one being that cadmium plating is a desirable rust preventive for springs under dynamic loading, but that zinc and tin platings are of little use.

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[3] President, Sam Tour & Co., Inc., New York, N. Y.

[4] Metallurgical Engineer, Mineral Dressing and Metallugy Div., Department of Mines and Resources, Ottawa, Ont., Canada.

This conclusion is not entirely in agreement with previous published work on other plated steels. Considering only the fatigue tests in air, it has been found in general that electrodeposited zinc has no effect on the fatigue strength of the base metal (Harvey 1930, Barklie and Davies 1930, Gough and Sopwith 1937), while some slight improvement has even been observed (Swanger and France 1932).[5] On the other hand, the few results reported on electrodeposited cadmium have indicated a reduction in fatigue strength (Gough and Sopwith 1937, Harvey 1932); unpublished tests have given 12 to 15 per cent reduction. Unfortunately no information is available to the writer concerning the effect of tin plating.

In seeking an explanation of this apparent reversal, some mention should be made of the possibility of internal stress in the deposited metal. It is known that, depending upon the plating procedure, metals can be electrodeposited with compressive or tensile stresses of considerable magnitude, a figure of 40,500 psi. tension having been reported for nickel. As normally deposited, cadmium and zinc coatings are internally stressed in compression, whereas tin is stressed in tension. These stresses, superimposed on those produced by the dynamic loading, would generally affect the fatigue strength, either favorably or adversely. Perhaps the author has some results which would indicate the type and magnitude of the stresses in the platings deposited under the particular conditions of his experiments. It is possible that the roasting treatment acted as a low-temperature anneal or stress-relieving treatment, though that would not account for the deleterious effect of the tin plating.

A more probable explanation would appear to lie in the phenomenon of hydrogen embrittlement. The author states that the pre-tempered high-carbon steel is quite susceptible to hydrogen embrittlement, and refers to the increased hydrogenation taking place with zinc and tin platings. Finally, from an examination of the fractures, he establishes the cause of the fatigue failures for these two platings as hydrogen embrittlement. It may be that the shot-peening operation rendered the steel more liable to embrittlement, since the well-known work of Zapffe and his collaborators has shown the increased susceptibility of cold-worked material. Zapffe has also stressed the importance of time and temperature on the recovery of plated steel from embrittlement, and it is possible that variations in these factors may account for the pronounced scatter reported in the fatigue tests on the zinc and tin plated springs.

In conclusion, the writer would like to ask whether the springs were examined for fatigue cracks during the progress of the tests, and where the majority of the failures occurred in relation to one end of the spring?

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[5] W. E. Harvey, "Zinc as a Protective Coating Against Corrosion Fatigue of Steel," *Metals and Alloys*, April, 1930, p. 458.

R. H. D. Barklie and H. J. Davies, "The Effect of Surface Condition and Electrodeposited Metals on the Resistance of Materials to Repeated Stresses," *Proceedings*, Inst. Mechanical Engrs., Vol. 1, p. 731 (1930).

H. J. Gough and D. G. Sopwith, "The Influence of the Mean Stress of the Cycle on the Resistance of Metals to Corrosion-Fatigue," *Journal*, Iron and Steel Inst., April, 1937, p. 293.

W. H. Swanger and R. D. France, "Effect of Zinc Coatings on the Endurance Properties of Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 430 (1932).

W. E. Harvey, "Cadmium Plating *versus* Corrosion Fatigue," "Pickling *versus* Corrosion Fatigue," *Metals and Alloys*, March, 1932, p. 69.

MR. JOHN R. GUSTAFSON (*author's closure by letter*).—Mr. Soderberg's comment regarding small amounts of nickel present in cadmium anodes is quite interesting, and could be a subject for considerable research. What are the effects of increasing amounts of nickel on the current density and the evolution of hydrogen? Many other questions arise which if answered would add to our knowledge of plating procedures and their effects. As a comment it also imposes a problem for future study.

Lead plating has been applied to springs. It was used on automotive water pump seal springs during the war as a substitute for non-ferrous materials. Porosity was a problem which was overcome by controlling the current density and increasing the thickness of the plate. Plating thicknesses exceeding 0.001 in. were required for satisfactory protection. Although I do not have any data pertaining to the effects of lead plating on dynamic stresses, I can report that lead-plated springs have given satisfactory service in the water pump application referred to after having been stored in bins for more than six months. This would indicate that lead plate, if properly deposited, will not cause hydrogen embrittlement or set up harmful residual stresses.

Mr. Ashkinazy asks a question which can be answered in generalities. Let me start first with the reasons for this investigation. Many people in the spring industry have picked up plated springs, deflected them just to be curious and had them break into two or more pieces. Initial investigations brought out the fact that pickling solutions used for cleaning puposes attacked the steel in such a manner that high localized stress concentrations, as well as a marked decrease in ductility, resulted. This was one of the causes for breakage of plated springs. A study of cleaning methods made it clear that the shot-peening and tumbling method was beneficial, in so far as increasing fatigue life was concerned. Therefore this cleaning method was used. After cleaning, the springs were immediately plated. The tests which took approximately four months to complete were carried out in the following sequence: (1) plain, (2) zinc, (3) tin, (4) cadmium. Since each group took approximately four weeks to test, twelve weeks had elapsed from the time of plating to testing the cadmium finish springs. Zinc had the advantage as far as time was concerned.

Mr. Tour's comments could be elaborated on for several hours, since they embody all methods of plating techniques. In the presentation of a paper it is difficult to determine every factor which will not only prove interesting, but also enliven the discussion. Mr. Tour's points of discussion could have been incorporated as a part of this paper had the "time factor" not been deemed important. This paper had as its objective the addition of knowledge, and some of it admittedly controversial, to the science of plating. To that end it serves a useful purpose.

To make an analytical study it is necessary to eliminate as many variables as possible and to adhere to standards that by their usage have proven satisfactory. For instance the thickness of plate has been commented upon. The plating thicknesses were arrived at by consulting specifications adopted by many industries as well as by several Government agencies.

Tests to determine suitable current densities for cadmium and zinc were completed before doing any work on the effects of these electro-deposited metals on springs. They were conducted in order to arrive at plating standards yielding uniform coverage, no burning, and no porosity. The optimum conditions determined from these tests were used. Tin plating was done in accordance with standards quite generally accepted by the plating industry.

Mr. Thurston's comments add additional ammunition to data presented. To compare findings of other investigators would also require that methods and materials be compared. Both are important in arriving at conclusions on the effects of electro-deposited coatings.

I wish I had accurate information on residual stresses of electro-deposited coatings, and fatigue testing offers one means of obtaining such data. The lower limit curves of Fig. 8 bring out the fact that compressive surface stresses were greater in the cadmium-plated springs than in the plain finish springs. These may have been due to the wet sand tumbling, the cadmium plate or a combination of both. The baking temperature after plating might also have been a contributing factor.

With reference to the comments on the effects of shot-peening, I feel that the results bring out the fact that this operation was beneficial in that fatigue life was definitely improved except for those cases where hydrogen embrittlement was a detrimental factor. Furthermore a strain-relieving operation followed the shot-peening in order to eliminate any adverse stress concentrations set up by this operation.

Each spring was examined for type and classification of fracture. Many fatigue fractures occurred at one and one quarter to one and one half coils from the tips of the ground ends. This agrees with findings of most investigators. The design of the spring, however, was such that the stress concentrations at the first portion of active material adjacent to the inactive ground coils were held to a minimum. Therefore, we obtained fatigue failures on every active coil. Failures due to hydrogen embrittlement occurred throughout the entire length of active material, setting no definite pattern. Some springs literally exploded, breaking into as many as four pieces. Examination of these failures brought out the fact that their origin might occur at any point in the active material, whereas the fatigue failures began at the inside diameter of the spring. Many springs which failed due to fatigue had incipient fatigue cracks adjacent to the point of failure.

I have tried to cover the issues brought out in the discussion. Where differences exist between my findings and those of other investigators, a comparison of the cleaning and plating methods should shed some light on the probable causes.

RATING OF EXPOSURE TEST PANELS

[EDITORS NOTE.—The following three papers were sponsored by Committee B-8 on Electrodeposited Metallic Coatings to give helpful information on examination and rating of outdoor exposure test specimens.]

RATING EXPOSURE TEST PANELS OF DECORATIVE ELECTRODEPOSITED CATHODIC COATINGS*

BY W. A. WESLEY[1]

SYNOPSIS

A system for rating the results of outdoor atmospheric exposure tests of chromium-plated finishes with a nickel or nickel-copper undercoat should be based solely on the appearance of the test panels. Appearance is difficult to define, and there is a great need of objective standards to lessen dependence upon the experience and cooperation of inspectors. As a step toward this goal a set of graphical reference standards was designed somewhat similar to that developed by the paint committee for rust resistance. A system of numerical ratings to go with these is recommended which is similar to that employed in the recent cooperative exposure tests of the National Bureau of Standards, the American Electroplaters' Society and the A.S.T.M. The new scale is set at a higher quality level because the behavior of coatings during the later stages of corrosion is considered of little practical interest. The paper is concluded with a discussion of the interpretation of inspection data.

The choice of rating system for evaluating the results of exposure tests depends upon the function of the coating under test. Decorative cathodic coatings suitable for outdoor exposure are limited practically to chromium-plated finishes with a nickel or copper-nickel undercoat. It will be agreed that the principal purpose in applying such finishes is to maintain unchanged the bright pleasing appearance of the newly prepared article. The inspector of exposure test panels is not concerned with the ability of the plated part to operate satisfactorily. Failure of a decorative coating to protect the basis metal from corrosion usually produces a displeasing appearance long before the attack has become severe enough to interfere with proper functioning of the part. The primary objective of the inspector should therefore be to evaluate the merit of the different finishes on the basis of time during which a satisfactory appearance is maintained. The behavior of coatings during later stages of corrosion is of little practical interest and should be ignored. It will be seen that rating systems used heretofore do not quite meet these goals.

SOME RATING METHODS PREVIOUSLY USED

Perhaps the earliest rating system in which a quantitative value was assigned in inspection records was that of Thomas and Blum **(1)**.[2] The numerical rating of a coating on a steel test panel was the number of weeks until the first appearance of rust visible at a distance of 1 m. (3.3 ft.). The reproducibility of this rating can hardly be good except for low values below 4 or 5. In addition it puts too much weight on the first evidence of rust, even though the appearance of the specimen may remain satisfactory long after this event. Later, Thomas and Blum (2) themselves concluded that this method of rating is unsatisfactory due to the difficulty of deciding what constitutes a slightly rusted condition and just when this first appears.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] Assistant Director, Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 813.

Baker and Pinner (3) employed a qualitative system for rating specimens in salt-spray corrosion tests which was later used with slight changes by other workers in recording the condition of outdoor atmospheric exposure specimens. Their system involved symbols representing five degrees of rusting, as follows:

P = perfect
VSR = very slightly rusted—close examination shows only traces of rust
SR = slightly rusted—close examination reveals distinct signs of rust
NR = noticeably rusted—moderately close inspection shows presence of rust
VNR = very noticeably rusted—rust plainly discernible at a distance of 3 or 4 ft.

They made an interesting attempt to assign to these symbols quantitative values which increased with successive periods of exposure to the salt spray. In actual spray tests there was a disappointing lack of agreement between the quantitative rating and the total time elapsed before the VNR stage was reached.

As part of a rather elaborate investigation of German automobile components, Bauer, Arndt, and Krause (4) carried on atmospheric exposure tests in which previously prepared standard samples were carried by the inspectors to aid in rating the condition of the exposed specimens. Three grades of attack were recognized, namely,

Alpha = Slight corrosion—tarnishing of the surface or slight local attack
Beta = Moderate corrosion—small patches of corrosion, staining or blistering, with no deterioration of the coating visible after removal of corrosion products.
Gamma = Disintegration of the coating or partial damage, remaining visible after removal of corrosion products.

Quantitatively, the only rating assigned at the end of a test was G = the number of weeks required for onset of "gamma" type corrosion.

Examination of the photographs in Bauer's book (4) leads one to conclude that many specimens did not reach the "gamma" condition until long after they had assumed an unpleasant appearance. Conversely, some were given a gamma rating due to a few local defects whilst they still retained a fairly pleasing appearance.

SYSTEM EMPLOYED IN COOPERATIVE TESTS

In the extensive atmospheric exposure tests cooperatively sponsored by the National Bureau of Standards, A.S.T.M., and the American Electroplaters' Society, a quantitative method of rating the condition of specimens was evolved by Blum and Strausser (5). Each inspector assigned to each specimen a numerical rating from 0 to 5 based upon the percentage of surface with rust or other defects. For coatings on nonferrous metals, all evidences of failure were taken into account, such as light or dark stains, blisters, cracks and peeling, to each of which equal weight was given. In some of the work steel specimens were rated separately as to (a) rust, and (b) all defects including rust, but the results indicated that the separate ratings were nearly alike for the types of coating tested.

The score for an inspection period was obtained by multiplying the average rating during the period by the number of weeks in that period. The total score for the test was then converted to a percentage score, that is, to the percentage of a perfect score for the total period of exposure involved. The relation of these numerical values is shown in Table I. The rating is seen to be an inverse function of the defective area.

TABLE I.—BLUM AND STRAUSSER RATING SYSTEM (5).

Proportion of Surface with Rust or Other Defects		Rating	Corresponding Score, per cent
Range, per cent	Average, per cent		
0	0	5	100
0 to 5	2.5	4	80
5 to 10	7.5	3	60
10 to 20	15	2	40
20 to 50	35	1	20
50 to 100	75	0	0

In actual application to thousands of specimens inspected over a period of six or seven years this system proved to be a practical one. The major conclusions drawn from the scores recorded in this project were undoubtedly correct. Nevertheless, there are two important ways in which the system can be improved:

1. *By Adopting Reference Standards.*—Appearance is a property of a test panel which involves psychological factors and is difficult to define. There is a great need for objective standards of appearance to aid the inspector.[3] It is questionable whether it is possible to estimate by simple inspection the percentage of area rusted on typical chromium-finished specimens except at the extremes near 0 and over 50 per cent. If the reader doubts this statement let him cover the captions attached to Figs. 2 to 6 and estimate by inspection alone the percentage of defective area in those figures. In the cooperative tests there was usually at least one "experienced" observer present at each inspection. This precaution was necessary and advisable to insure uniformity in defining each of the five ratings.

2. *By Raising the Scale of Ratings.*—During the last series of exposures in the 1936 Cooperative Tests, E. M. Baker suggested to Blum and Strausser (5) that, in addition to the numerical ratings, each inspector should indicate whether in his opinion the specimens were still satisfactory as judged from the standpoint of a user. Most inspectors reported as "unsatisfactory" those specimens with a rating of 3 or less. The author agreed with these inspectors and believes that ratings below 3 had no practical significance and that the zero of the rating scale should have been raised to correspond to the quality represented by a rating of 3.

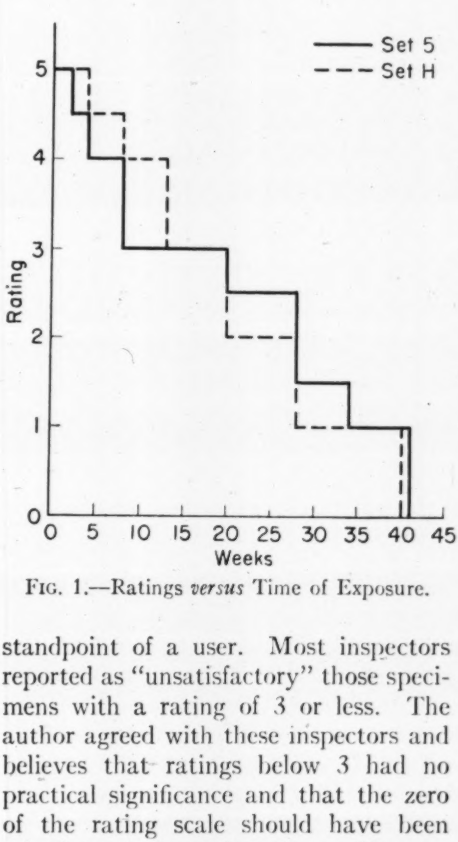

FIG. 1.—Ratings *versus* Time of Exposure.

The argument can be presented graphically. The solid lines in Fig. 1 show the change in rating for set 5 of the 1932 series of Cooperative Tests exposed at New York (6). An average rating greater than 3 was shown by this set for only 8 weeks while the exposure was continued for about 41 weeks to give a final percentage score of 51. Let us suppose that another set of specimens at this location behaved as indicated by the dotted lines in Fig. 1. The final per-

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[3] See Standard Method for Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces (D 610 - 43), 1946 Book of A.S.T.M. Standards, Part II, p. 1186.

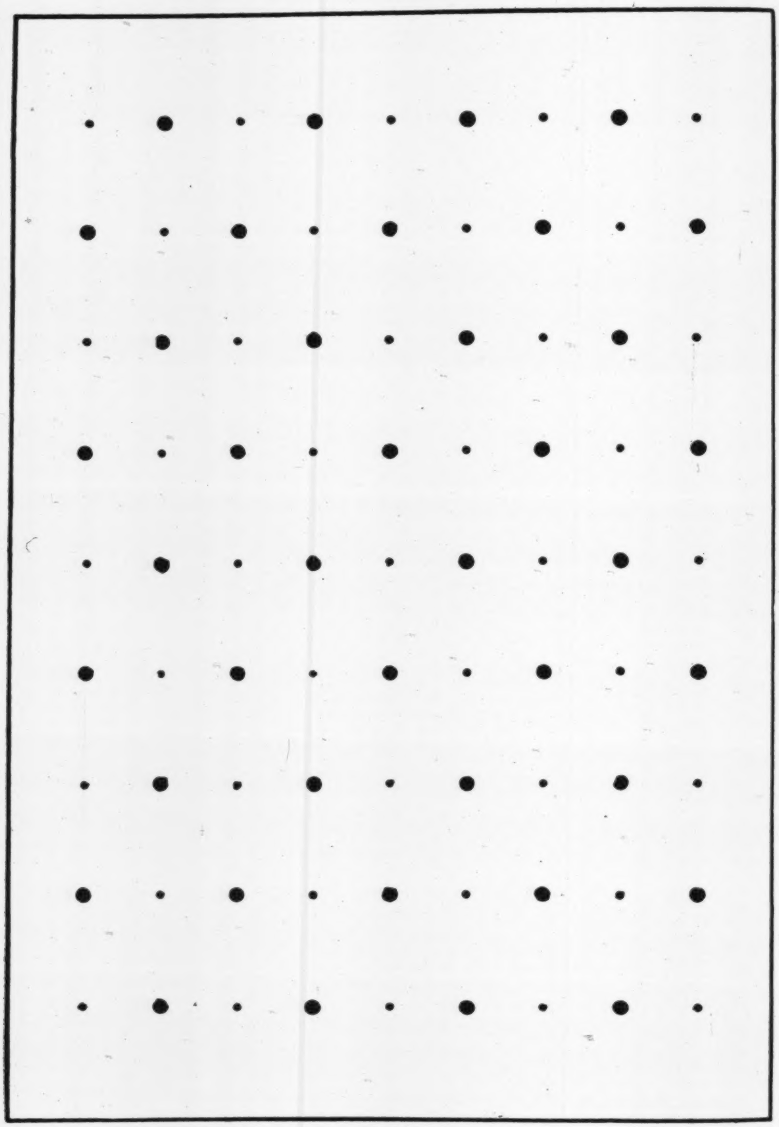

FIG. 2.—Unsatisfactory Standard.

Defective surface area, 1.0 per cent, 80 spots per panel (4 by 6 in.)

centage score of this hypothetical set H would have been the same as that of set 5. From the practical or users' point of view, set H was very much superior to set 5 since it retained a satisfactory appearance (rating better than 3) for 13 weeks. If a scale with zero at the level of rating 3 had been used, the scores for set H would have been almost double that for set 5.

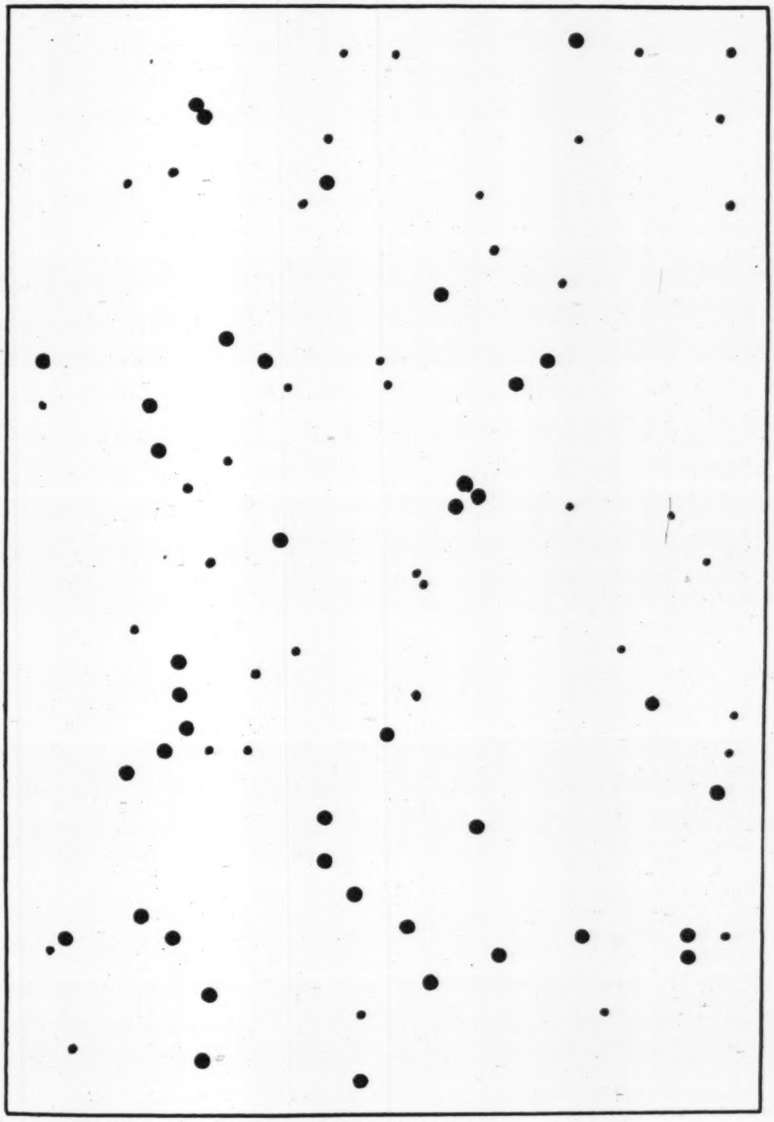

FIG. 3.—Standard Rating No. 1.
Defective surface area, 1.0 per cent, 80 spots per panel (4 by 6 in.)

RECOMMENDED RATING SYSTEM

The author believes that the Blum and Strausser rating system can be modified to advantage in the following ways:

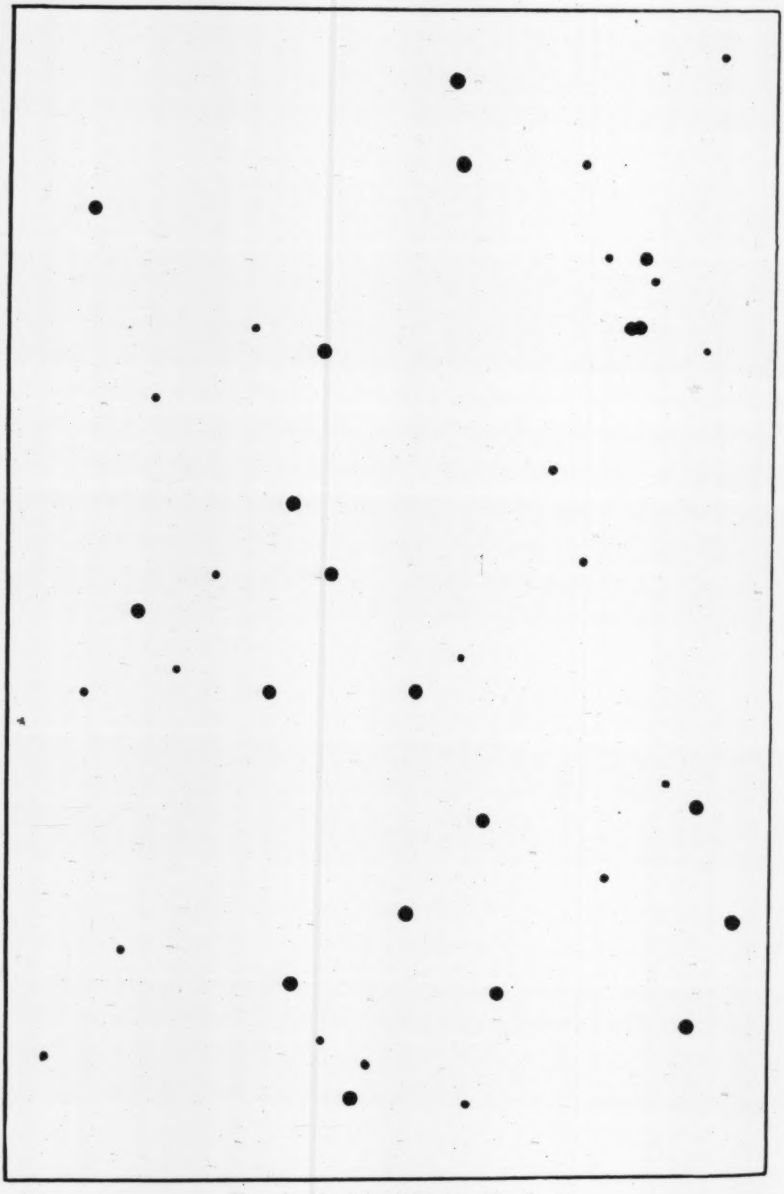

FIG. 4.—Standard Rating No. 2.
Defective surface area, 0.5 per cent, 40 spots per panel (4 by 6 in.)

Reference Standards:

Graphical reference standards to aid in visual examination of specimens have been adopted by the A.S.T.M. for estimating the degree of rusting of

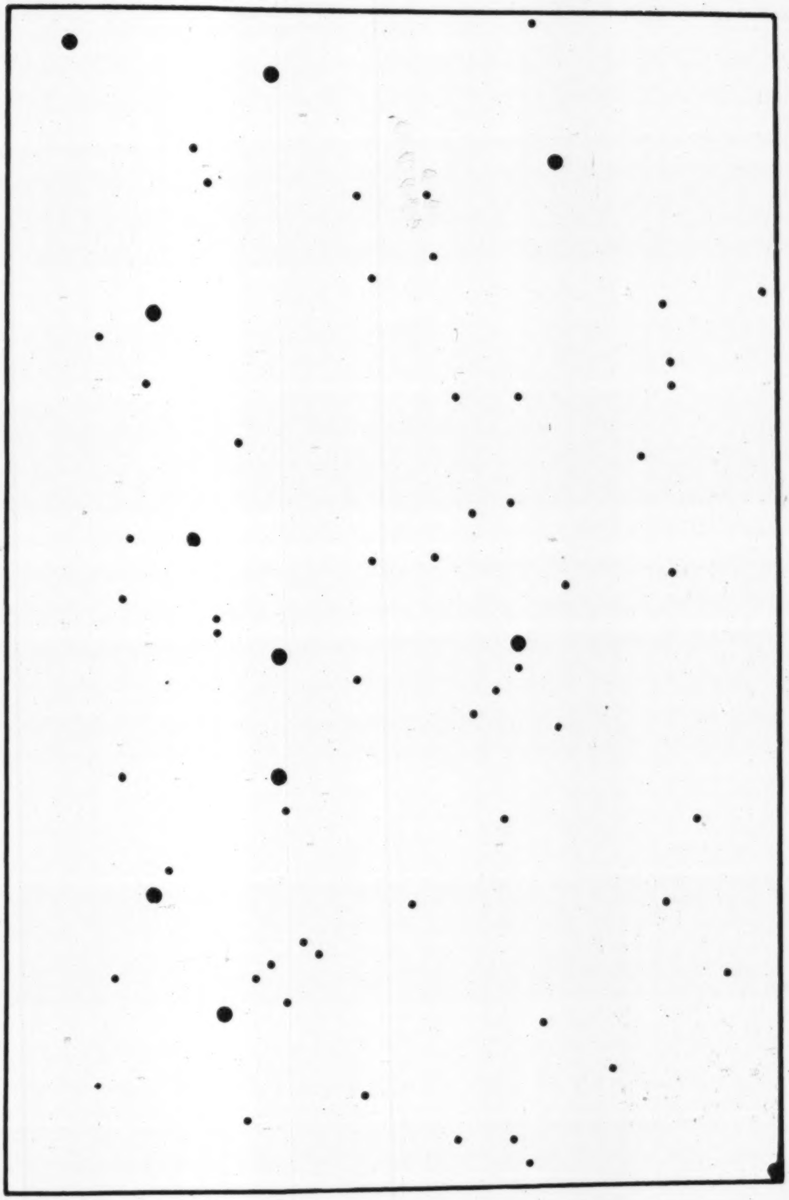

FIG. 4*a*.—Alternate Standard Rating No. 2.
Defective surface area, 0.5 per cent, 66 spots per panel (4 by 6 in.)

painted iron and steel surfaces,[3] for evaluating the microstructure of graphite in cast iron[4] and for classifying steels according to grain size.[5] If one attempts to prepare a chart representing an exposed chromium-finished panel with

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[4] Tentative Recommended Practice for Evaluating the Microstructure of Graphite in Gray Iron (A 247 - 41 T), 1946 Book of A.S.T.M. Standards, Part I-A, p. 1073.

[5] Standard Classification of Austenite Grain Size in Steels (E 19 - 46), 1946 Book of A.S.T.M. Standards, Part I-A, p. 682.

1 per cent of its area rusted or otherwise defective it is quickly evident that no regular pattern of spots resembles even remotely the appearance of actual corroded panels. A chart such as Fig. 2 is of little use as a reference standard.

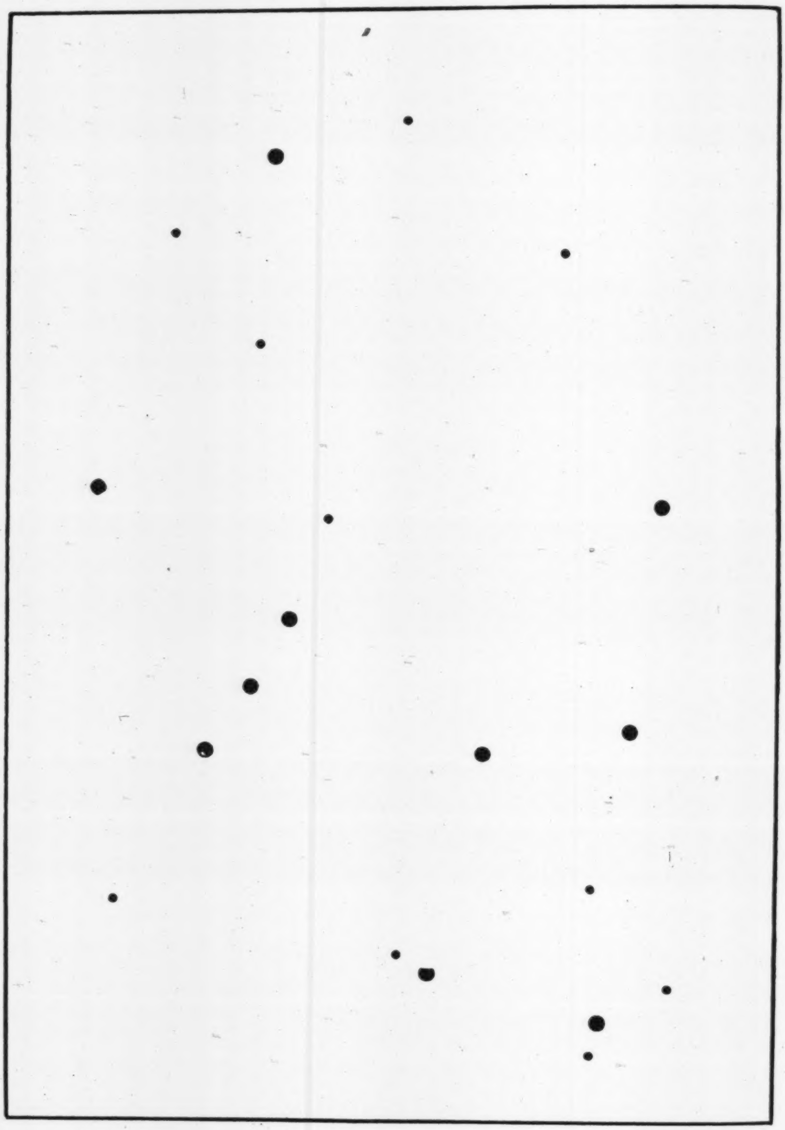

FIG. 5.—Standard Rating No. 4.
Defective surface area, 0.25 per cent, 20 spots per panel (4 by 6 in.)

The characteristic distribution of defective spots on exposure panels has been observed by the author to be a random one. If the same number of spots and total defective area as those in Fig. 2 are used, but with random distribution of the spots, a chart like Fig. 3 is obtained. This was done by dividing a 6 by 4-in. rectangle into 10,000 equal rectangular subdivisions with the aid of cross-section paper. Then 40 spots of 2-mm. diameter were placed at locations represented by the first 40 four-place numbers of a table of random numbers, and 40 spots of 1-mm. diameter were plotted at locations represented by the next 40 numbers drawn from the table (7). In the same way Figs. 4, 5, and 6

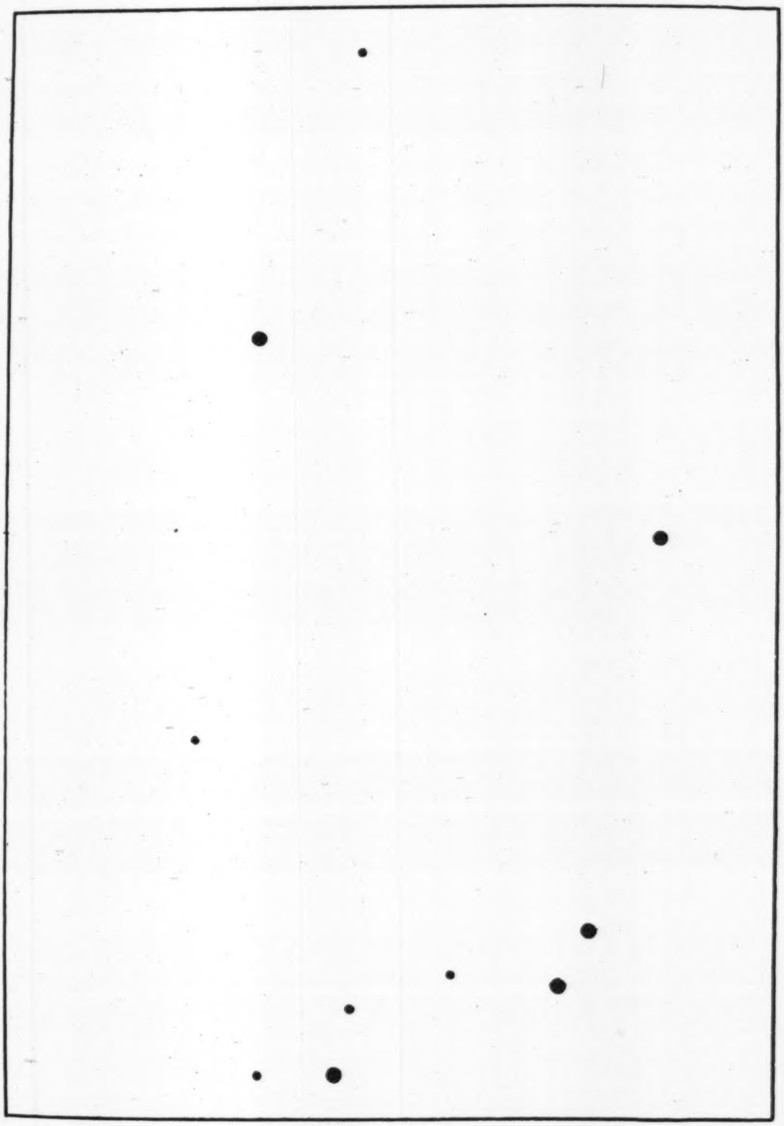

FIG. 6.—Standard Rating No. 8.
Defective surface area, 0.125 per cent, 10 spots per panel (4 by 6 in.)

were prepared using other but fewer numbers from the table. Numbers representing positions within $\frac{1}{4}$ in. of the edges of the panel were in all cases discarded.

B. B. Knapp has suggested to the author that certain types of failure of exposure panels are better represented by use of a greater proportion of small spots. He submitted as an example Fig. 4*a* for comparison with Fig. 4.

Rating:

Ratings assigned to the reference standards are inversely proportional to the defective area except at rating 10:

Rating	Defective Area, per cent
1	1
2	$\frac{1}{2}$
3	$\frac{1}{3}$
4	$\frac{1}{4}$
5	$\frac{1}{5}$
6	$\frac{1}{6}$
7	$\frac{1}{7}$
8	$\frac{1}{8}$
9	$\frac{1}{9}$
10	0

This relationship places a premium on quality in the range $\frac{1}{4}$ to 0 per cent defective area without penalizing too severely for failure of the coating at just one or two points per panel.

It will be noted by persons experienced in the cooperative tests that the standard for rating 1 is approximately the same in *appearance* as the old rating 3. The proposed scale therefore starts at a considerably higher level of quality.

The value of a decorative coating is lost as soon as it ceases to be decorative. All defects which detract from the pleasing appearance of the test panel should therefore be included in assigning a rating for its condition. A brief statement of the kinds of defects noted and considered in selecting the rating should form part of the inspection data. In comparing a given panel with the standard reference charts the inspector should attempt to base his judgment on general appearance and should not attempt to estimate the actual percentage of defective area.

Defects within $\frac{1}{4}$ in. of the edges of a panel are ignored.

PRESENTATION OF INSPECTION DATA

Percentage scores are an aid in evaluating the relative merits of different coatings exposed at the same time and place. They should be calculated as before by multiplying the average rating during each inspection period by the number of weeks in that period, adding these products to obtain the total score, and finally converting the total score to a percentage score, that is, to the percentage of a perfect score for the period of exposure selected. At the same time the original data showing the ratings for each inspection should also be presented for the following reason: Some users of plated parts may wish to use a scale of ratings even higher in quality than the present scale. In a particular application a condition like Standard Rating 4 may arouse customer complaints. If he has available a set of standard charts and actual rating-*versus*-time data anyone can make his own set of percentage scores at the quality level desired for a specific service.

CLEANING EXPOSURE PANELS

Blum and Strausser made some attempts to learn the effect of cleaning dust and soot from exposure panels before inspection (**5**). Due to the difficulty of applying a cleaning procedure uniformly in the field, they did not advocate any regular treatment of this kind. When dust and soot are present to an extent seriously interfering with inspection they should be removed with the aid of water and a soft cloth. The use of an abrasive in this treatment is open to question because this would remove not only dust and dirt but also patches of corrosion product. Corrosion products of iron and copper are undoubtedly capable of accelerating corrosion of the coating over which they spread. Thus, any differences due to a difference in nature of the corrosion products developing upon them would be masked by thorough cleaning.

A return to the position formerly used in mounting exposure panels, namely, at 45 deg. with the horizontal, would probably decrease the rate of accumulation of soot and reduce the need for cleaning treatments.

INTERPRETING INSPECTION DATA

The ultimate purpose in making atmospheric corrosion tests of decorative coatings is to improve coating methods and structures so that coated parts will retain longer a pleasing appearance in simple atmospheric service. No one seriously expects ordinary atmospheric exposure tests to indicate the correct order of merit of a series of coatings for automobile bumpers if the bumpers in question are going to be used in a city where calcium or sodium chloride is frequently spread on the highways. Special tests must be developed for this purpose. It is not safe to assume even that the order of merit indicated by tests in a sulfurous atmosphere will hold for a coastal atmosphere, although the major trends disclosed in the cooperative tests (5) were amazingly consistent from one atmosphere to another.

Subject to these kinds of limitations, great reliance can be placed upon the results obtained with panels exposed to the same atmosphere at the same time. Blum and Strausser (5) concluded from a study of the thousands of ratings made in the Cooperative Tests that the final percentage scores were reproducible within ± 10 per cent. This seems to be consistent with general experience in corrosion testing so that there is no reason to be dissatisfied with the reliability attainable in exposure tests of plated coatings. It is anticipated that the use of reference standard rating charts will improve the situation by aiding persons who have not seen the exposure panels to understand and interpret the inspection data, and by facilitating comparison of results of tests run by different groups.

Some unfavorable criticism has been voiced of the use of percentage scores in the Cooperative Tests as the basis for comparing panels, but the author agrees with C. H. Sample (8) that this procedure is valid and the fairest way of correlating results from tests at different locations or tests of different duration. For comparing a series of coatings exposed at the same time and at one location either the total scores or percentage scores can be used; the results are the same. Both these numbers take account of the length of time during which a given panel maintains a given rating.

The actual ratings of coated panels and time of each inspection should be included in the published data in addition to the scores for the use of those who wish to compare individual coatings at the end of some arbitrarily selected period of time and for those who wish to compile scores at a higher quality level.

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(3) E. M. Baker and W. L. Pinner, "Protective Value of Chromium Plate," *Journal*, Soc. Automotive Engrs., Vol. 22, p. 331 (1928).

(4) O. Bauer, H. Arndt, and W. Krause, "Chromium Plating with Special Reference to Its Use in the Automobile Industry," translated by E. W. Parker, Edward Arnold & Co., London (1935).

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(7) R. A. Fisher and F. Yates, "Statistical Tables," Oliver and Boyd, Edinburgh (1938) Table 33.

(8) C. H. Sample, "Inspection and Rating of Test Panels Employed in the Outdoor Exposure Tests of Electroplated Coatings," *Monthly Review*, Electroplaters' Soc., Vol. 32, p. 23 (1945).

DISCUSSION

Mr. C. H. Sample.[1]—The problem of rating exposure test specimens of the decorative cathodic type, and the attendant problem of reporting the results in terms useful to others, has long confronted finish engineers and research people. I say both finish engineers and research people because the latter in general are largely interested in comparing the corrosion behavior of different coatings, whereas the former are primarily interested in the performance (with respect to time) of particular coatings in particular environments.

In my opinion Mr. Wesley's paper is an outstanding contribution toward the solution of the problem, and on behalf of Committee B-8 I extend our appreciation for his efforts. His demonstration of the random occurrence of points of failure in electro-deposited coatings is particularly noteworthy.

With the thought that Wesley has not intended the rating system he described, to be the final, perfect system for all purposes, I should like to offer a few comments and suggestions:

First, I believe we will all agree that some reference standards, which the inspectors can use for direct comparison in the field, are highly desirable. These may be either of the type Mr. Wesley has prepared or actual photographs of suitable specimens. I would suggest however, that rather than rate the specimens on the basis of an arbitrary rating scale of, say, 10 to 1 or zero, the estimated per cent of the area stained or otherwise defective be recorded. This would permit a direct tie-in of exposure results on decorative coatings, on which a relatively small amount of corrosion is objectionable, with results on other types of coatings where protection of the basis metal, rather than appearance, is of primary importance.

I believe we will all agree with Mr. Wesley's opinion that decorative coatings can be considered unsatisfactory when in the aggregate corrosion spots or stains on their surfaces constitute one or more per cent of the surface area. I believe, however, that because of possible misinterpretation, it is not desirable to give a specimen with only 1 per cent of its area defective a rating of zero.

Actually for many purposes, and especially if it is cleaned periodically, a specimen which would be given a rating of 1 or zero on Mr. Wesley's scale, will exhibit a considerably extended useful life.

I am of the opinion that any final rating scheme we may adopt for decorative cathodic coatings should include a periodic rating of specimens from which accumulated stains and corrosion products have been removed. Admittedly, such cleaning may not be necessary for research work in which the corrosion behavior of different coatings of the same general type are being compared. However, I do believe it is necessary if one object of the test is to provide reference data on the behavior of particular coatings for the benefit of finish and design engineers. Admittedly to include cleaning in any inspection system introduces

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[1] The International Nickel Co., Inc., New York, N. Y.; Chairman of Committee B-8 on Electrodeposited Metallic Coatings.

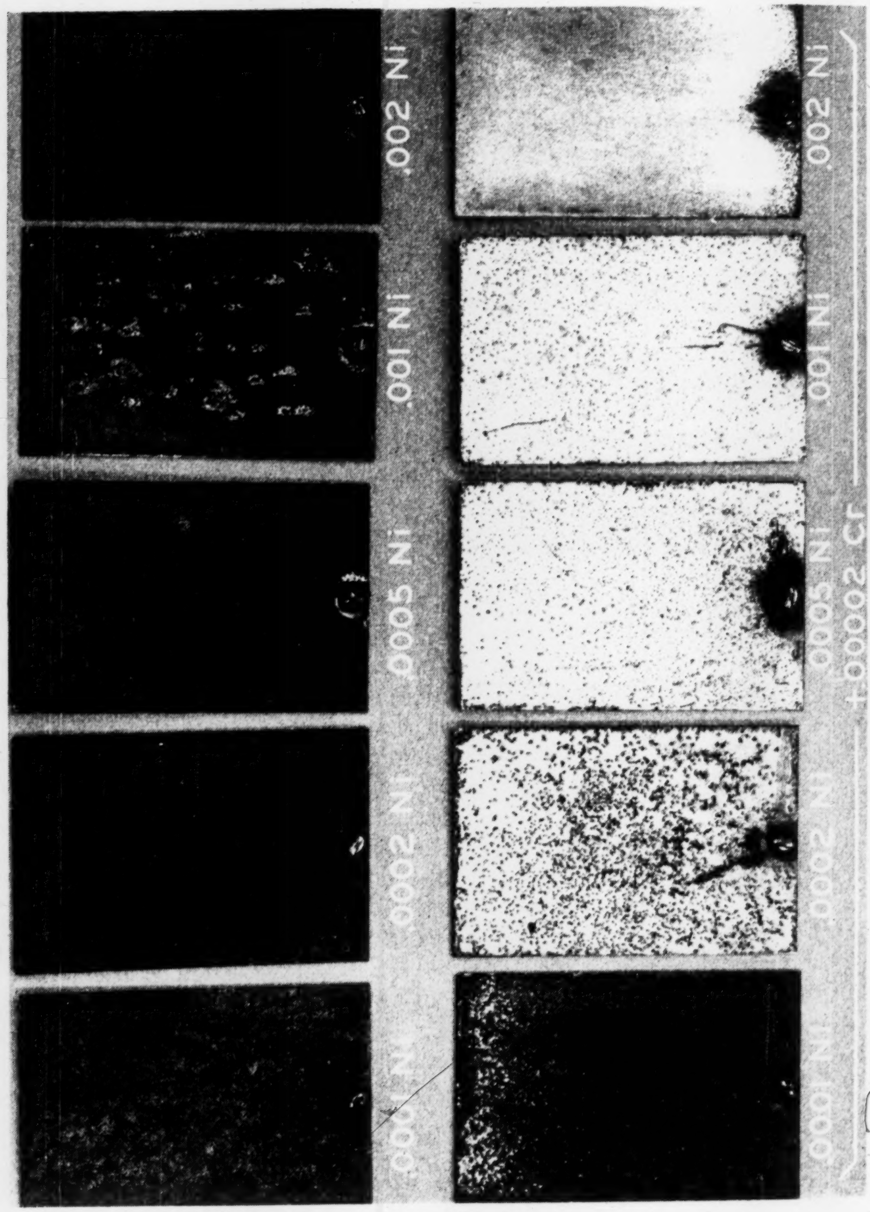
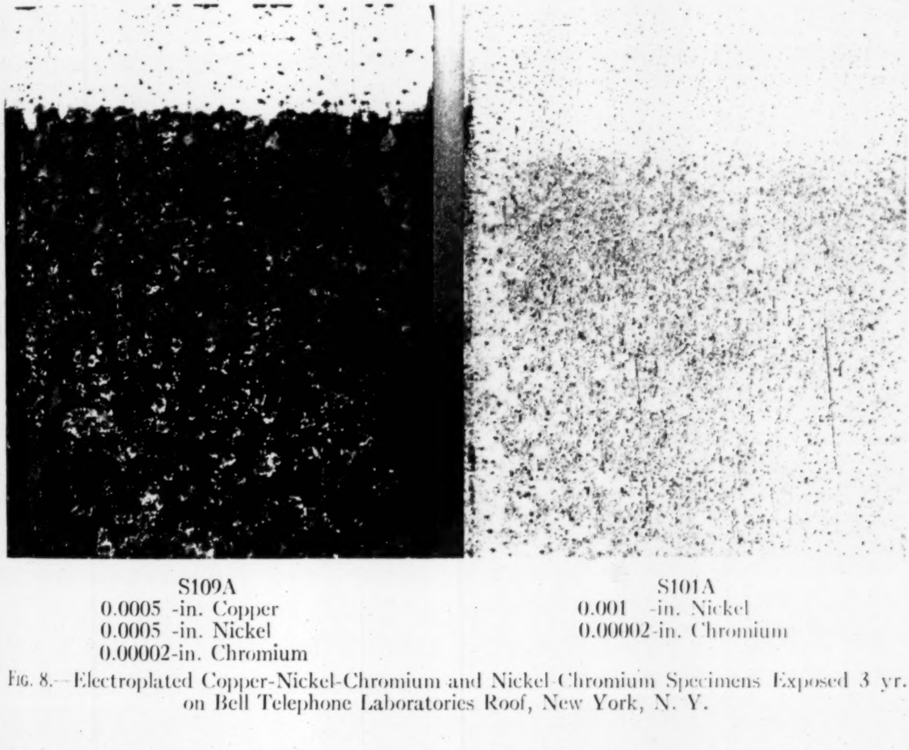

.002 Ni
.001 Ni
.0005 Ni
.0002 Ni
.0001 Ni
.002 Ni
.001 Ni
.0005 Ni
.0002 Ni
.0001 Ni
+.00002 Cr

Fig. 7.—Electroplated Nickel and Nickel-Chromium on Steel Exposed on Bell Telephone Laboratories Roof, N. Y., N. Y., May, 1943 to January 1945.

additional problems involving the standardization of the cleaning procedure, etc. However, in the interest of completion and over-all value of the results, I believe we should give the matter further attention. Actually it would appear that most of the problems could be solved simply by providing sufficient specimens to allow for cleaning a few of them periodically, others of course to remain uncleaned.

The accompanying Fig. 7 shows typical nickel and nickel-chromium plated steel specimens with increasing thicknesses of nickel (0.0001 to 0.002 in.) which were exposed approximately 18 months on the roof of the Bell Telephone Laboratories. We are concerned here primarily with the nickel-chromium deposits and, I believe, on the basis of Mr. Wesley's rating scale, all of them would be rated as zero. However, I believe it will be agreed that for some purposes there is still a lot of useful life left in at least three, and possibly four of the coatings shown at the bottom (0.0002, 0.0005, 0.001, and 0.002 in. of nickel, respectively).

Figure 8 shows copper-nickel-chromium and nickel-chromium plated specimens of the same total thickness (0.001 in.) which were exposed out of doors in New York for three years.



S109A
0.0005 -in. Copper
0.0005 -in. Nickel
0.00002-in. Chromium

S101A
0.001 -in. Nickel
0.00002-in. Chromium

FIG. 8.—Electroplated Copper-Nickel-Chromium and Nickel-Chromium Specimens Exposed 3 yr. on Bell Telephone Laboratories Roof, New York, N. Y.

The top inch of each has been cleaned with a mild abrasive. I believe it will be agreed that, while the appearance of the uncleaned surfaces is very bad, they can be cleaned and, for some purposes, may be quite satisfactory.

Figure 9 shows nickel and nickel-chromium coatings (0.0001 and 0.0002 in. nickel) on brass, which have been exposed to the New York City atmosphere for about 18 months, and as might be expected are badly stained. The bottom half of each specimen has been cleaned with a mild abrasive. Undoubtedly, some corrosion of the brass has occurred. Nevertheless, it is possible to remove the stain, and, when kept clean, the coating will continue to serve a useful purpose for some time.

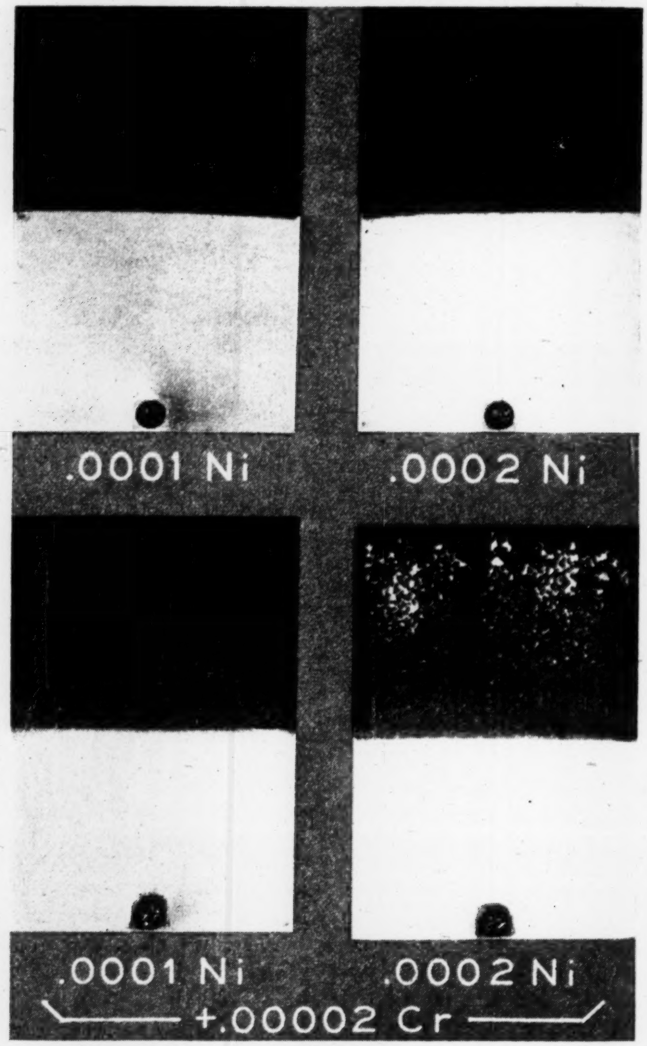


FIG. 9.—Electroplated Nickel and Nickel-Chromium on Brass Exposed on Bell Telephone Laboratories Roof, N. Y., N. Y., May, 1943 to January, 1945.
Bottom Half of Specimens Cleaned.

MR. H. A. PRAY.[2]—I am in hearty support of Mr. Sample's point of view, and wish to remark that the exploratory copper-nickel-hromium atmospheric tests being conducted at Pittsburgh by Subcommittee II of Committee B-8, where the specimens have been out perhaps a year and a quarter, also point in the same direction. Simply dipping some of those panels, that appeared to be 100 per cent rusty, in acid to dissolve the rust stain that was on the surface of the nickel, resulted in panels that would be rated "four" by Mr. Wesley's scheme.

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[2] Battelle Memorial Institute, Columbus, Ohio.

I think there may be a parallel between the behavior and evaluation of the copper-nickel-chromium coatings and the tarnishing of silver alloys in tableware service. So far as I know, they are all subject to tarnish. Some tarnish more rapidly than others They have a tremendously long useful life in service because it is fully realized that a certain amount of polishing is required.

The zero to ten rating proposed by Mr. Wesley should certainly reveal fundamental differences between coating systems, and the application of the random distribution principle to the establishment of rating standards appears to be perfectly sound. However, I cannot believe that the proposed system will tell us the actual useful life of decorative coatings which in service are normally subject to a reasonable amount of cleaning and polishing.

MR. R. J. MCKAY.[3]—On comparing the viewpoint expressed by Mr. Wesley with those set forth in the papers by Messrs. Pray[4] and Soderberg,[5] I note a substantial difference in the purpose of rating different samples which results in differences in evaluating appearance factors. Taking these three classes of coatings which have been assigned for the several papers; we have first the nickel-chromium coatings, which must primarily retain their appearance and secondarily protect and are cathodic to the base metal; second we have lead which is also cathodic and is used to protect the base metal, the appearance being unimportant, and, third, zinc and cadmium whose purpose is to give protection by anodic action. These coatings are thus very different in their appearance and in their purpose. And I question whether there can be a completely uniform method of rating for all of them, or at least, an invariable standard. While we might possibly use the same yardstick to measure them all, although even that is questionable, certainly we will have to use different points along our measuring stick as the points of failure.

MR. W. A. WESLEY (*author's closure*).—Mr. McKay stated the objection to expanding the rating system into the higher per cent defective range better than I can. One way in which we could satisfy both purposes, I think, is to assign numerical ratings related to the percentage area rusted, as I have suggested, and then continue to evaluate the panels beyond that stage by simply recording the percentage rust. Anyone interested in purely decorative coatings (and there is certainly a much greater application of nickel and chromium in the purely decorative field than in all the engineering applications combined) will be able to compare exposure results from the aesthetic point of view while those interested in the ability of the part to continue its function can go on to make comparisons based on larger areas rusted.

There was a slight misunderstanding of what I said with regard to cleaning. I think that cleaning without use of abrasives is permissible and will not change the relative order of merit of coatings of this type.

(*By letter*) I wish to thank Messrs. Sample, Pray and McKay for their thoughtful comment. The importance of the topic is well realized by Subcommittee II of Committee B-8 in its current series of exposure tests. I

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[3] Chemical Engineer, Nickel Sales Dept., The International Nickel Co., Inc., New York, N. Y.

[4] H. A. Pray, "Inspection of Exposure Test Panels with Nondecorative Electrodeposited Cathodic Coatings," see p. 822.

[5] Gustaf Soderberg, "Inspection of Exposure Test Panels with Anodic Electrodeposited Coatings," see p. 826.

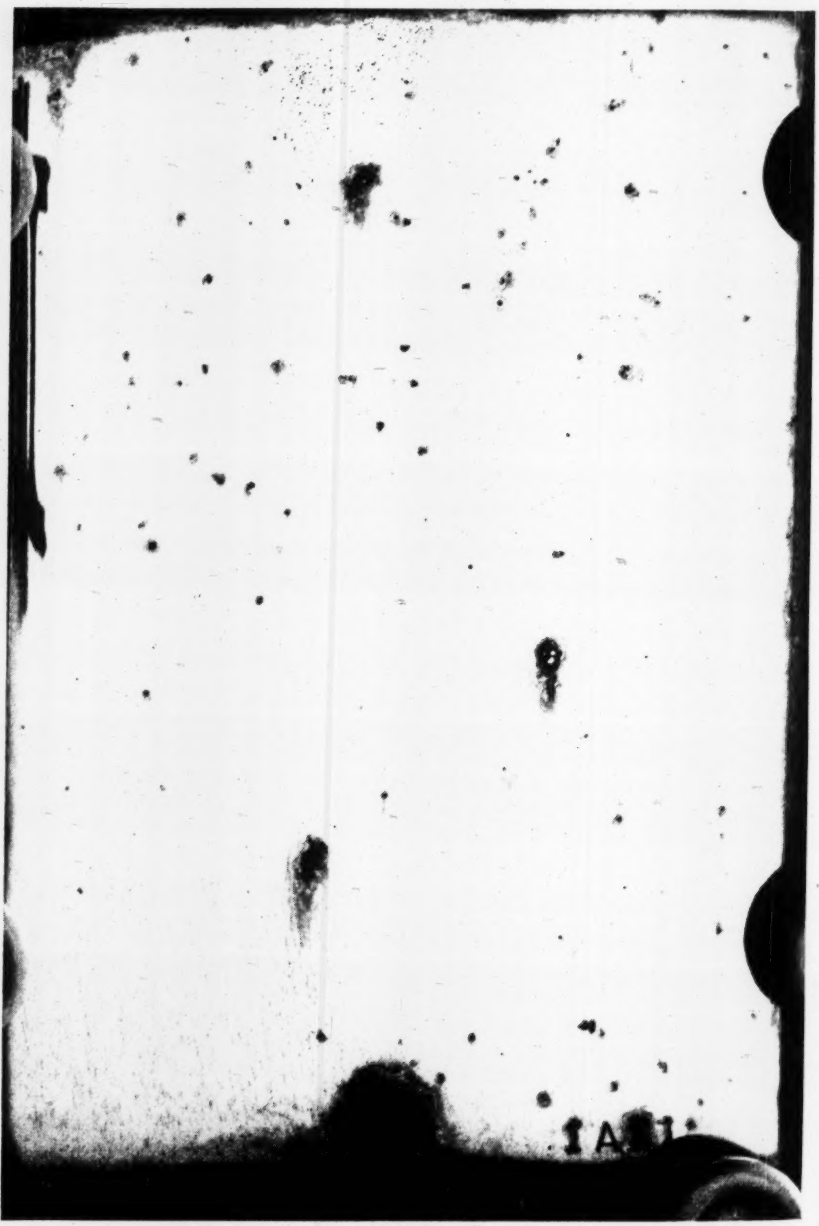

FIG. 10.—Shows Actual Exposed Nickel and Chromium-Plated Steel Panel Selected to Correspond to a Standard Rating of ½ per cent Surface Defective.

hope this committee will lead the way in establishing graphic or photographic standards which can then be used not only in its own work but in the activities of the American Electroplaters' Society Research Projects and in industrial research laboratories. Figure 10* is offered as a photograph of an actual exposed nickel and chromium-plated steel panel selected to correspond to a standard rating of $\frac{1}{2}$ per cent surface defective. This is an example of photographs which could well be used as inspection standards.

In re-reading the paper and its discussion I note that no comment has been made regarding descriptive records of the nature of failure and condition of exposure panels. It would not be wise to discontinue making such notes during inspection even if rating standards are used. They are necessary to aid in interpreting the numerical ratings and in explaining abnormalities which arise in all work of this sort.

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* Kindly furnished by Mr. F. L. LaQue.

INSPECTION OF EXPOSURE TEST PANELS WITH NONDECORATIVE, ELECTRODEPOSITED CATHODIC COATINGS*

By H. A. Pray[1]

Synopsis

The function of cathodic, electrodeposited metallic coatings used for purely protective purposes is discussed and the way in which they deteriorate on weathering is described. Rating systems are summarized, with particular emphasis on the methods used for the exposure tests of electrodeposited lead coatings on steel made by Subcommittee II on Performance Tests of A.S.T.M. Committee B-8 on Electrodeposited Metallic Coatings. The information given is intended to be helpful to the evaluator who needs to form an independent judgment of the utility of a coating from a study of reported inspection records.

Function of the Coatings

The primary purpose of the coatings under consideration is to prevent or retard deterioration in the structural properties of the base metal to which they are applied. Although appearance is a psychological consideration in practically any product, it is of relatively minor importance as compared to the protection afforded the base metal by coatings in this classification. Maintenance of integrity of the functional properties of the structure being protected is the important criterion in determining the utility of a nondecorative, cathodic, protective coating. Such a coating fulfills its function to the extent that it prolongs the time period during which the structure can be used for its intended purpose.

Steel, being the most commonly used structural metal and being prone to relatively rapid deterioration when subjected to weathering, is the base material most often requiring protection. A number of the metals which can be electroplated on steel may be classified as nondecorative, protective coatings. Of these, the interest of Subcommittee II on Performance Tests of A.S.T.M. Committee B-8 has recently been centered in electroplated lead. The discussion will, therefore, be concerned most directly with lead coatings, although the general principles involved apply broadly to metallic coating systems of similar protective mechanism and purpose.

Atmospheric Behavior of Electroplated Lead on Steel

Two factors appear to control the protective life of lead coatings on steel during atmospheric exposure: (1) the rate of weathering of the electroplated lead comprising the coating which in turn depends upon the exposure conditions, and (2) the thickness, uniformity, porosity, and continuity characteristics of the deposit. Both appear to be important.

The first noticeable corrosion effect on lead-plated panels exposed to the atmosphere is the accumulation of a mottled, white corrosion product, which is independent of the base metal or the thickness of the coating. This is evidence of atmospheric attack, on the lead itself, and is related to the (presumed) sulfur dioxide content of the particular atmosphere. Additional weathering results in the appearance of small pinhole rust spots which are indicative of coating porosity. On further exposure, the small rust spots increase in size and number. In the course of time, "bleeding" from the pores spreads the appearance of rust over the surface of the coating, although, on by far the greater part of the panel area, the lead is still intact, adherent, and protective. Finally, corrosion of the base metal reaches a stage at which the coating is destroyed and takes on the appearance of rusted bare steel over part or all of the area. The beginning of this phase marks the first significant deterioration of the base metal itself. The end of this stage obviously marks the end of the time during which the coating exerts any influence over the rate of corrosion of the steel.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] Battelle Memorial Institute, Columbus, Ohio.

Although visual inspection cannot give a quantitative measure of the protective value of a coating system, it is possible to obtain indications and trends, and a relative idea of the probable usefulness of different coating systems. The objective of a visual inspection method should be to provide a description of a given panel at a given location and exposure time that will enable a reader who has not seen the test panels to arrive at a reasonably accurate evaluation of the condition of the coating and, in so far as possible, the condition of the base metal. Quantitative values, defining the extent of damage to the steel, can be obtained from measurements of weight and strength losses suffered by the steel itself. Quantitative values, defining the corrosion rate of electrodeposited lead, can be obtained from measurements of the weight loss suffered by the coating prior to any attack on the steel. But whether the inspection method is based on qualitative visual examination or quantitative weight and strength loss determinations or both, it should preferably not involve an arbitrary definition of "failure." A condition that might be considered "failure" in one application might be called satisfactory performance in another.

RATING SYSTEMS IN PREVIOUS USE

Reports of atmospheric corrosion tests on electroplated lead coatings are not numerous. Clarke (**1**),[2] in reporting the results of exposure tests at Woolwich, England, describes the panels variously as "rusted all over," "few very small rust spots," "small rust patches," "one third of surface rusted," etc., and makes no estimate of the extent of damage to the steel base.

Soderberg (**2**), reporting results on fluoborate lead-plated steel panels exposed at Detroit, estimates the percentage of the panel surface rusted.

DuRose (**3**) discusses the relative protective value of electrodeposited lead and lead-tin alloys on steel. He reports the results of both salt-spray and atmospheric tests. The salt-spray data indicate in some cases the hours to "breakdown" and in others, the number of pinholes and percentage of surface rusted. The outdoor exposure data are reported as "months to failure," "failure" being defined as "1 to 2 per cent rust."

Diggin (**4**) considers failure to be the time to the first appearance of rust in salt-spray tests.

Subcommittee VIII on Field Tests of Metallic Coating[3] of Committee A-5 on Corrosion of Iron and Steel has been conducting an extensive series of exposure tests on coated wire and wire products, including a few hot-dipped, lead-coated wires and similar coatings on hardware.[4] The conclusions are being based on: (1) visual examination, including (*a*) qualitative descriptions of appearance (metallic, gray, yellow, brown, etc.) and (*b*) quantitative estimates of "the percentage of the area that has actually rusted and the percentage that is yellowed, seemingly by iron corrosion products, but has not yet shown actual rusting of the base metal;" (2) tension tests; and (3) loss-of-weight tests.

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[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 825.

[3] Now (1947) Subcommittee XV—Ed.

Quoting from the 1939 report of Committee A-5,[4] "It is difficult to judge the extent of actual corrosion of the base metal. A better seasoned judgment will be provided by future tests on the tensile strength of removed specimens." The tabulated data in the reports referred to indicate that no distinction is being made between the appearance of rust that results from "bleeding" from minor uncoated areas (pinholes) and the loose rust that appears at the time that serious degradation of the base metal occurs. Thus, in the 1943 report,[4] a lead-coated (1.96 oz. per sq. ft.) steel wire exposed at Sandy Hook, N. J., for 4.45 yr. is rated 90 R + Y and at 4.90 yr., 95 R + Y (95 per cent rusted and/or yellowed).

THE PRESENT SYSTEM

The inspection method used by Subcommittee II of Committee B-8 for electroplated lead on steel represents an attempt to record and describe the progressive deterioration of the coating from its weight loss; of the steel, from weight and tensile loss; and of the base metal plus coating system by visual examination. It differs in certain details from the Committee A-5, Subcommittee VIII system, as will be evident from the following description:

Visual Examination:

The number of pinhole rust spots, visible at normal reading distance, are counted, unless they exceed 25, and recorded as 3P, 10P, 18P, etc. When the number exceeds 25, the counting becomes laborious and the condition is described as "MP," many pinholes—too many to count or estimate. Very little judgment is required in obtaining this part of the record.

The percentage of the area of the panel that appears to have been affected by iron corrosion products, whether it be the smooth discolored area that is the result of "bleeding" from pinholes or the flaky rust that is the result of actual attack on the base metal, is recorded as 10S, 30S, 75S, etc. Roughly, 10, 30 or 75 per cent of the panel surface is either stained or rusted. The number is either an eye estimate or is determined with the aid of a transparent guide, appropriately ruled.[5] This part of the record requires a certain amount of judgment, but agreement among experienced inspectors is fairly good.

The percentage of the area of the panel covered with flaky red rust is then estimated and recorded as 5R, 20R, 60R, etc. This represents roughly the area on which the coating has ceased to be protective and whose initial appearance indicates the first real damage to the steel. The distinction between "S" and "R" is not easy to make, since the transition is gradual and the difference is one of degree. It is particularly difficult to make on panels exposed in an industrial atmosphere such as New York. However, the distinction is of considerable significance in defining qualitatively the protective value of the coating. Those who have been active in the inspection of the panels agree that an estimate of "R" should be made as precisely as possible.

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[4] Report of Wire Inspection Committee on Field Tests of Wire and Wire Products, *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 156 (1939); Vol. 41, p. 101 (1941); Vol. 43, p. 78 (1943); Vol. 45, p. 70 (1945).

[5] See Gustaf Soderberg, "Inspection of Exposure Test Panels with Anodic Electrodeposited Coatings," p. 826, this volume.

A set of standards would be extremely useful to the inspector and more especially to the interpreter of the recorded data, to whom this difference between "S" and "R" is difficult to describe in words. Black and white roductions are not useful for this purpose. Actual panels, representing the different stages of weathering, are not available. Color transparencies are excellent, but their general distribution presents problems. So far, it has been necessary to rely largely on the collective judgment of the inspection group.

The interpreter who has not participated in the inspection of the panels should be able to obtain a fairly good picture of the condition of the coating and some idea of the relative protectiveness of the various coating systems from the recorded results of the visual inspections. A panel reported as "5P" is in significantly better condition than one reported as "MP–10S." One described as "MP–10S" is superior to one given an "MP–80S–2R" rating. The latter is entering the stage of measurable damage to the base metal. At a rating of "100R," the interpreter can infer that the protective life of the coating has ended.

Again, the results of visual inspection are qualitative, at best. The recorded results are gratifyingly regular. The discrepancies which do occur must be attributed to the inevitable "judgment" factor on the part of the inspectors.

Weight Loss of the Coating:

The inspection system includes an examination of the coating itself by provision for periodic determinations of the weight loss of panels prior to significant attack on the base metal. A quantitative measure of the rate of weathering of the electrodeposited lead is thus obtained. It has some significance in the estimation of the protective life of a coating, particularly in the case of the thicker coatings. In the case of thin coatings, the weather resistance of the coating metal is overshadowed by nonuniformity and porosity effects.

Weight and Strength Loss of the Steel Base:

The system also includes an examination of the steel base by provision for periodic determination of both weight loss and loss of tensile strength. Since the purpose of electroplating with metals of the type under consideration is primarily to provide protection to the base on which it is plated, the direct examination of that base must provide the final basis for estimation of the protective value of the metal.

More emphasis has probably been placed in this discussion on the visual inspection evaluation of the condition of the base metal than its importance justifies. This is the case largely because it requires more explanation than do the more direct and quantitative methods. An interpreter of the data can be guided by the reported results of visual inspection. His final conclusions will more closely approach fact if they are based on the more exact results of weight and tensile strength determinations of the condition of the steel base.

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(3) A. H. DuRose, "The Protective Value of Lead and Lead-Tin Deposits on Steel," *Transactions*, Electrochemical Soc., Vol. 89, p. 417 (1946).

(4) M. B. Diggin, "Lead Plating," ***Metal Finishing***, Vol. 41, p. 418 (1943).

[See discussion of Wesley paper, page 815.]

INSPECTION OF EXPOSURE TEST PANELS WITH ANODIC ELECTRODEPOSITED COATINGS*

By Gustaf Soderberg[1]

Synopsis

The corrosion of zinc or cadmium-plated outdoor exposure specimens proceeds in two stages, an initial stage during which minute rust spots may be found which may be significant in corrosion-fatigue resistance, and a final stage during which increasingly large areas are denuded of plate and exhibit rusting. The rusting during the former stage may be evaluated simply by counting the spots, during the latter by placing over the panel a shield of clear plastics ruled so as to facilitate counting the percentage of rusted area. The whole panel should be evaluated, including the edges, because the second stage usually begins at the lower edge. The extent of sacrificial protection may be judged by the width of the band of bare metal between the rusted and still coated areas. This paper deals specifically with the inspection of electrodeposited coatings of zinc and cadmium on steel, exposed out-of-doors. It is believed that other nondecorative anodic coatings behave in the same general manner when exposed out-of-doors, and that the same general principles of inspection and evaluation will apply to them.

The prime function of this type of coating in outdoor exposure is to provide corrosion protection to the base metal—appearance is of little or no consequence.

Published Inspection Methods

Published inspection methods may be divided into two groups: visual methods exemplified by the work by Committee A-5 on Corrosion of Iron and Steel (**1**)[2] by Blum *et al.* (**2**), and by Clarke (**3**); and weight-loss methods such as employed by Patterson (**4**), and by Hippensteel and Borgmann (**5**).

In the early A.S.T.M. ferrous hardware tests (**1**), zinc and cadmium coatings were inspected visually, and the specimens were rated on the basis of the percentage of rusted area. Being especially familiar with the inspection of hot-dipped zinc coatings, the inspection committee distinguished between red rust—which is considered to emanate from the base metal—and yellow or orange rust—which in the case of hot galvanized coatings is considered to form on the corrosion of the iron-zinc alloy layers. Since no such layers occur in electroplated coatings, the distinction is believed artificial.

Blum *et al.* (**2**) employed a system in which the specimens were rated from time to time by visual observation. A surface with 0 per cent rust was given the rating of 5; 0 to 5 per cent rust, 4; 5 to 10 per cent rust, 3; 10 to 20 per cent rust, 2; 20 to 50 per cent rust, 1; 50 to 100 per cent rust, 0. The intermediate ratings reported are the averages of the ratings of the several inspectors. Blum *et al.* multiplied the average rating during the inspection period by the time elapsed between inspections to obtain the "score" for the period. The scores for all the periods were added together and divided by the perfect score, that is five times the total time elapsed, to obtain a "percentage score." The relative value of two coatings after a given period of time was judged by their relative "percentage scores."

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Consulting Engineer, Graham, Crowley and Associates, Jenkintown, Pa.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 830.

Clarke (3) in reporting on indoor corrosion tests simply uses expressions like "almost free from rust," "rust-colored patches," etc.

Patterson (4) determined weight losses after removal of corrosion products but did not state the method employed in their removal.

Hippensteel and Borgmann (5) in their tests on plated 0.0008-in. thick zinc and cadmium coatings on steel followed the progress of corrosion by weighing the plates at intervals of two months. Prior to weighing, half of the specimens were scrubbed with a stiff bristle brush under water, the other specimens were only lightly brushed with a soft "varnish" brush. Since the cleaning methods did not materially affect the measured rates of corrosion, it was considered reasonable to assume that neither tended to disturb the metal itself and that in either method practically all of the corrosion products were removed. Passano (6), comparing the relative weight losses of zinc and cadmium with the relative protective value in the same atmosphere as determined by visual inspection, concluded that the weight-loss measurements give reliable indications.

Each one of these methods suffers certain drawbacks as shown in the following.

THE EARLY STAGE

The development of rust on specimens with zinc or cadmium coatings proceeds in two stages. During the initial stage, the corrosive attack is centered upon the coating itself which corrodes rather uniformly with discoloration. Minute rust spots may appear at discontinuities at any time. As a rule they grow in number as the coating becomes thinner and may increase in size toward the end of this stage. Sometimes the rust spots disappear for a time, presumably having been washed off by a violent rain.

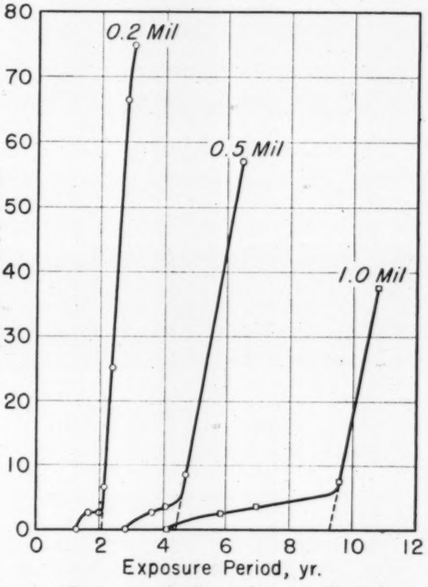
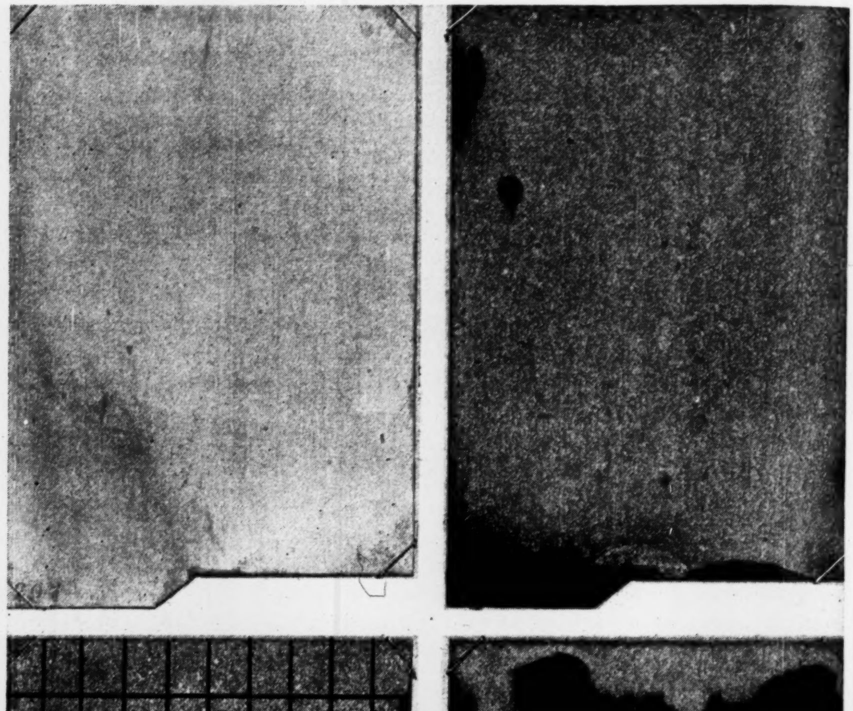


FIG. 1.—Progress of Corrosion of Cadmium-Plated Steel Panels Exposed in Washington, D. C. Intercepts on abscissa indicate time for substantial rusting.

The total percentage of area taken up by such rust spots is very small at any time and cannot be easily estimated by the eye. The rust would be noted by Clarke (3) but not by Committee A-5 (1). With the Blum *et al.* (2) rating system, it would lower the rating of the panels from 5 (0 per cent rust) to 4 (0 to 5 per cent



Unexposed

2 per cent rust

32 per cent rust with ruled plastic sheet

89 per cent rust

FIG. 2.—Progress of Corrosion of Steel Panels Plated with 0.0002-in. Zinc and Exposed in Washington, D. C.

rust). In the reconversion from rating to percentage of rust used in the preparation of Fig. 1, the early values are undoubtedly too high.

The question arises: How much attention should be paid to these early stages of base-metal corrosion? They are not important as an appearance factor as in the case of Wesley's (11) decorative cathodic coatings or as an indication of substantial pitting as in Pray's (10) nondecorative cathodic coatings. On stripping the coating without attacking the steel, one will observe in some instances a very slight etching below the rust spot but never any deep corrosion pit. It is well known that pitting does not substantially affect ultimate strength in tension and has to be quite pronounced before it affects elongation values. Hence, although direct experimental data are lacking, one can safely assume that corrosion of steel through pores in zinc or cadmium coatings will have little if any effect on the static tensile quality of the steel.

TABLE I.—COATING THICKNESS *versus* ENDURANCE LIMIT.

Type Steel	Coating	Endurance Limit, psi.		Protection,[a] per cent	Reference
		in air (S_a)	in water (S_w)		
Heat-treated....	None	70 000	18 000		(7)
Cr-V steel.	0.0002 in. Cd	...	35 000	33	
	0.0004 in. Cd	...	42 000	46	
Heat-treated....	None	60 500	20 000		(8)
S.A.E 1045......	0.00022 in. Cd	...	42 000	54	
Heat-treated.....	None	60 500	20 000		(9)
S.A.E. 1045.....	0.0011 in. Zn	...	60 500	100	

[a] Percentage protection = (S_w coating − S_w bare)/(S_a bare − S_w bare).

The situation may be different when the plated object is subjected to both corrosion and alternating stresses. The meager data in Table I indicate that thin coatings furnish a lesser percentage protection than thick coatings. Thin coatings are also the ones which show the most rust spots. Hence, the porosity may be an important factor in corrosion fatigue, and a record should be kept of it.

The method of recording rust spots can be very simple. As a rule the spots are very small and few in number until just before the second stage sets in, and a simple count of the number appears sufficient without any attempt to translate the number into percentage of rusted surface.

THE FINAL STAGE

During the final stage of exposure, a different mechanism of rusting appears. The coating has dissolved faster on certain areas than on others, usually on the lower portion of the test specimen to which moisture adheres the longest, and finally the steel is bared in part and shows substantially continuous areas that have rusted. This is shown in Fig. 2 taken from the report by Blum *et al.* (2). The rusted areas are best estimated by placing over the surface a sheet of clear plastic which has been ruled into 100 areas of equal size, each one representing 1 per cent of the area to be judged (Fig. 2). The number of areas which cover rust only are counted. Certain areas cover both rusted and unrusted portions; the rusted portions are estimated and added to the previous figure.

In the past, estimates of percentage of rust have often been made without the use of such a guide. An inexperienced observer is likely to make rather large errors, especially when the rusted areas are unevenly distributed.

The Blum *et al.* (2) rating and scoring systems may have certain value in the *evaluation* of the results. However, it is felt that the original data, percentage of surface area rusted, used by Committee A-5 (1), should go into the *report of results*.

Some data taken from Blum *et al.* (2) are shown in Fig. 1. The ratings have been converted to percentage of rusted area and hence are approximate. By extending the almost straight, nearly vertical portion of the curves to the abscissa, a fair measure of time for substantial rusting may be had and reported. It is believed to be a better measure of rust protection than the time for 50 per cent rusting as has been proposed.

EDGE EFFECTS

The question of whether the whole surface of the specimen should be judged or one-quarter of an inch around the edges should be disregarded, needs some consideration. The fact that, depending on the racking during plating, the coatings on the edge may be thicker or thinner than on the remainder of the surface would lead one to play safe and disregard the rusting nearest the edge. Against this stands the observation mentioned above that substantial rusting is likely to start from the bottom edge. With thick deposits or deposits in a relatively noncorrosive atmosphere, the progress of extension of the rusted area may be slow. Hence, if the lower edge is disregarded, the time for the beginning of substantial rusting may be delayed a great deal and the coating may be said to protect against substantial rusting considerably longer than it actually does. It is submitted that the most reliable results are obtained if the rusting of the edges is taken into account. This, of course, requires that the specimens be plated with the same thickness of coating on the edges as on the major portion of the surface. It disregards the slight acceleration of corrosion which sometimes occurs on sharp corners.

EXTENT OF SACRIFICIAL PROTECTION

As rust spreads across a specimen, it will be noticed that there is a band of bare unrusted steel between the rusted and still coated areas. The width of this band is a measure of the sacrificial protection afforded by the coating to the steel in the particular atmosphere at the time of observation and may well be noted in the report.

CONCLUSION

Methods have been presented for the inspection and evaluation of test panels with nondecorative anodic coatings. They are purely visual as in the case of decorative cathodic coatings and, unlike the methods for nondecorative cathodic coatings, do not require the use of mechanical testing for confirmation.

REFERENCES

(1) Report of Subcommittee VIII of Committee A-5, *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part I, p. 149 (1929); Vol. 31, Part I, p. 184 (1931); Vol. 33, Part I, p. 156 (1933); Vol. 35, Part I, p. 92 (1935).

(2) W. Blum, P. W. C. Strausser, and A. Brenner, "Corrosion-Protective Value of Electrodeposited Zinc and Cadmium Coatings on Steel," *Journal of Research*, Nat. Bureau Standards, Vol. 16, pp. 185–212 (February, 1936). (*RP 867.*)

(3) S. G. Clarke, "The Atmospheric Corrosion Resistance and Protective Value of Metallic Coatings," *Journal*, Electrodepositors' Tech. Soc., Vol. 15, p. 141 (1939).

(4) W. S. Patterson, "The Resistance of Electrodeposits to Corrosion, with Special Reference to Cadmium and Zinc," *Journal*, Electrodepositors' Tech. Soc., Vol. 5, p. 91 (1930).

(5) C. L. Hippensteel and C. W. Borgmann, "Outdoor Atmospheric Corrosion of Zinc and Cadmium Electrodeposited Coatings on Iron and Steel," *Transactions*, Electrochemical Soc., Vol. 58, p. 23 (1930).

(6) R. F. Passano, "The Harmony of Outdoor Exposure Tests," Symposium on the Outdoor Weathering of Metals and Metallic Coatings, Washington Regional Meeting, March 7, 1934, p. 49. (Issued as separate publication STP No. 17.)

(7) D. J. McAdam, Jr., "Fatigue and Corrosion-Fatigue of Spring Materials," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 51, p. 55 (April, 1929).

(8) W. E. Harvey, "Cadmium Plating vs. Corrosion-Fatigue—Pickling vs. Corrosion Fatigue," *Metals and Alloys*, Vol. 3, pp. 69-73 (March, 1932).

(9) W. E. Harvey, "Zinc as a Protective Coating against Corrosion Fatigue of Steel," *Ibid.*, Vol. 1, No. 10 (April, 1930).

(10) H. A. Pray, "Inspection of Exposure Test Panels with Nondecorative Electrodeposited Cathodic Coatings," p. 822, this volume.

(11) W. A. Wesley, "Rating Exposure Test Panels of Decorative Electrodeposited Cathodic Coatings," see p. 803, this volume.

[See discussion of Wesley paper, page 815.]

SYMPOSIUM ON MEASUREMENT OF ENTRAINED AIR IN CONCRETE

INTRODUCTION

By A. T. Goldbeck[1]

It seems to be the general consensus that in recent years no single development in the technology of concrete has approached in importance that of air entrainment. As experience was gained with air entrained concrete it became evident that the quantity of entrained air was subject to variation due to a number of influences and, further, that variations in air content resulted in variations in the strength and durability of the concrete. It became highly important that exact and convenient methods for measuring air entrainment be developed. One of the pioneers in this endeavor was J. C. Pearson, as the result of whose efforts the Society issued a Tentative Method of Test for Air Content (Volumetric) of Freshly Mixed Concrete (C 173 - 42 T).[2] Then it was decided that our Standard Method of Test for Yield of Concrete, C 138 - 39, could also be used to calculate the air content in concrete and so the title was changed to read, Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138 - 44).[3]

For a while it was thought that the problem of air measurement had been settled, but soon other methods were tried, the Indiana Method, the Rolling Method and the Klein-Walker Method, —all of them aimed at greater certainty or more convenience, particularly for use under field conditions.

It has seemed desirable to bring these various methods into the light to compare them for the degree of accuracy to be expected, their convenience, reliability and speed of operation to the end that the Society might select one or more of them for standardization.

In the present Symposium the various methods that have thus far been tried are described and in several cases comparisons are made and thus the reader is furnished with data which will permit him to decide which method he should adopt for his particular purpose.

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[1] National Crushed Stone Assn., Washington, D. C.; Chairman of Symposium Committee.

[2] 1946 Book of A.S.T.M. Standards, Part II, p. 1411.

[3] *Ibid.*, p. 513.

PROCEDURES FOR DETERMINING THE AIR CONTENT OF FRESHLY-MIXED CONCRETE BY THE ROLLING AND PRESSURE METHODS[*][1]

By Carl A. Menzel[2]

Synopsis

This paper describes apparatus and methods for determining the air content of fresh concrete based on two different principles, neither of which requires weighing scales. These methods are:

1. *Rolling Methods*, direct volumetric methods in which the entrained air is removed by rolling the concrete in an excess of water and is indicated directly by the volume of liquid required to restore the original liquid level,
2. *Pressure Method* (proposed by W. H. Klein and Stanton Walker) in which the volume of entrained air is indicated by the change in volume of the concrete upon application of a known pressure.

Field and laboratory tests show good correlation between the two methods. The paper includes proposed tentative methods based on the extensive studies which were made to develop the proper apparatus, calibration and testing technique for making the methods as practical and dependable as possible under job conditions.

Introduction

Determinations of the air content of fresh concrete have usually been made by the so-called "gravimetric method" (A.S.T.M. Designation: C 138 - 44)[3] in which the sum of the absolute volumes of the ingredients in a known volume of concrete is calculated and subtracted from the known volume, the difference being taken as the volume of air in the concrete. It has been found that this method is not as accurate, rapid, or practical as desired for field determinations of air content. Moreover, the method requires a sensitive weighing scale (sensitive to about 0.01 lb.) which is difficult to transport and maintain under job conditions.

With the wider use of air-entraining cements and concretes, the need for more dependable and practical methods for measuring air content in both the field and laboratory has become acute. To meet this situation, extensive studies were made of methods based on the following two different principles for measuring air content, neither of which requires weighing scales:

1. *The Volumetric Principle* by which the volume of air is measured directly, and
2. *The Pressure Principle* by which the volume of air is measured indirectly by the change in volume it undergoes when subjected to a known pressure.

The studies resulted in the development of apparatus and procedures for conducting tests for air content by the rolling method, or by the modified rolling method (methods based on the volumetric principle) and the pressure method (based on the pressure principle). Comparative tests showed good agreement between the results obtained by the rolling and pressure methods with each other and with the results by the gravimetric method.[4]

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[*] Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] Prepared under the auspices of A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

[2] Senior Research Engineer, Research Laboratories, Portland Cement Assn., Chicago, Ill.

[3] Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content" (Gravimetric) of Concrete (C 138 - 44), 1946 Book of A.S.T.M. Standards, Part II, p. 513.

The apparatus and procedures which will be described are based on both laboratory and field studies. These studies developed the testing technique required to make the methods of test as practical and dependable as possible under various job conditions. The proposed procedures (Parts I and II) are based on the accumulated field and laboratory experiences to date with the methods.

PART I.—GENERAL DESCRIPTION OF APPARATUS AND METHODS

ROLLING METHOD

The rolling method of test is based on the volumetric principle for measuring air content. A sample of freshly-mixed concrete is placed in a closed vessel and the entrained air is removed primarily by rolling the concrete in an excess of water. The volume of air that escapes (usually as a finely divided thick foam) is indicated by the volume of liquid (isopropyl alcohol) that has to be added to restore the original level of water over the concrete to a reference mark in the neck of the flask.

Apparatus:

The general appearance of the equipment developed by the Research Laboratories of the Portland Cement Assn. for conducting the rolling and modified rolling tests is shown in Figs. 1 and 3.

Operation of Rolling Method:

The operation will become clear from the diagrams of Figs. 1 and 2 and the remarks which follow. The round-bottom, flanged steel bowl, nominally 8 in. in diameter and 8 in. deep has a capacity of approximately 0.22 cu. ft. of concrete when struck off flush with the top flange. After placing of the concrete by the usual rodding procedure, a second bowl of the same size having a neck provided with a reference mark (a machined shoulder on the inside of the neck) and a quick-closing cap is then clamped to the bowl containing the concrete, thus forming a metal flask of a constant volume. The flanges of the two bowls bear against a rubber gasket making a watertight seal.

Water is then added over the concrete[5] to fill the flask to the reference mark on the neck of the upper bowl and the cap fastened in place. A light portable cradle with small rollers is then used to move the flask from the vertical position Fig. 2(*a*) to the inclined position in Fig. 2(*b*).

In the position shown in Fig. 2(*b*), the flask is slowly rotated on the rollers of the cradle while the upper end and sides are vigorously tapped with a rawhide mallet. This gradually shifts the concrete mass from the upper to the lower end of the flask and causes small increments of the concrete to be tumbled or cascaded into the water (Fig. 2(*c*)), releasing the entrapped air and foam from the concrete mixture (Fig. 2(*d*)). After about ten revolutions the flask is inclined in the opposite direction (Fig.

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[4] Carl A. Menzel, "Development and Study of Apparatus and Methods for the Determination of the Air Content of Fresh Concrete," *Journal*, Am. Concrete Inst., Vol. 18, No. 9, May, 1947, pp. 1053–1072; *Proceedings*, Am. Concrete Inst., Vol. 43, p. 1053 (1947).

[5] To avoid eroding the concrete with a jet of falling water, a funnel with a closed perforated spout is used to spray the water horizontally to the wall of the flask without agitating the concrete.

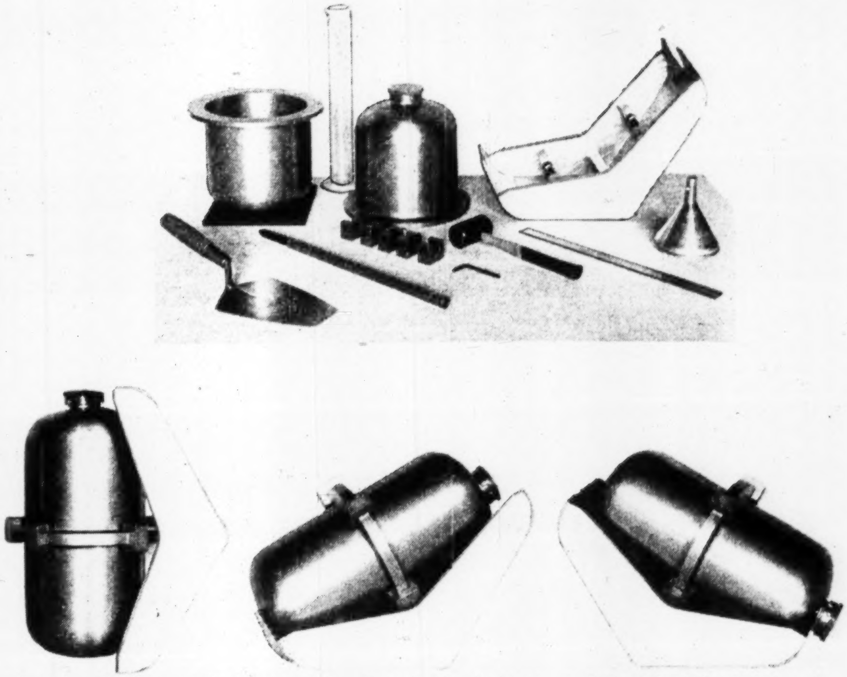

FIG. 1.—Equipment for Determining Entrained Air Content of Freshly-Mixed **Concrete** by the Rolling Method.

When filled, the 8 by 8-in. bowl in upper view accommodates a 0.22-cu. ft. sample of concrete. Lower views show use of cradle to tilt assembly and permit rolling in inclined positions.

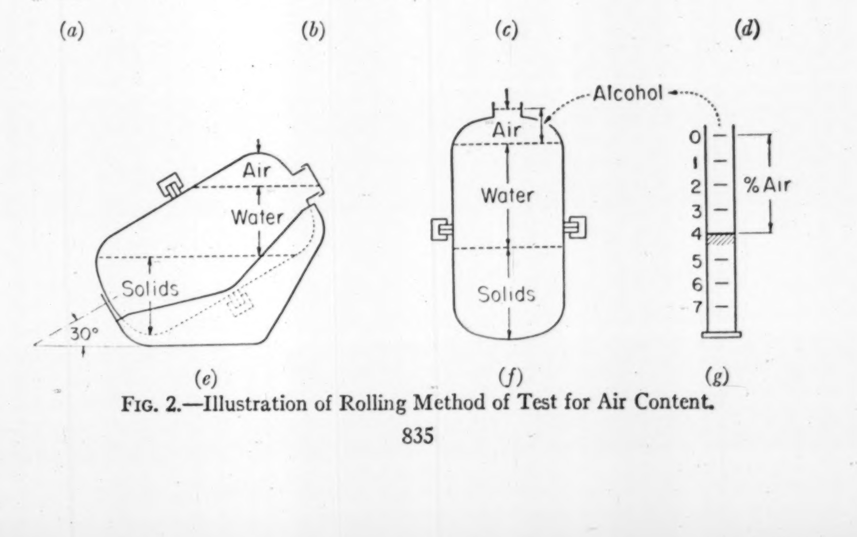
Zero mark
Water
Concrete
(a)
Concrete
Water
30°
(b)
Tumbling action
Rollers
(c)
Air
Water
Solids
(d)
Air
Water
Solids
30°
(e)
Alcohol
Air
Water
Solids
(f)
0
1
2
3
4
5
6
7
% Air
(g)

FIG. 2.—Illustration of Rolling Method of Test for Air Content.

2(*e*)) and more air is removed by rolling and tapping while the concrete is shifted to the opposite end of the flask. After about ten revolutions at this inclination the flask is returned to its original upright position (Fig. 2(*f*)) and isopropyl alcohol[6] is added from the graduate to destroy the foam and bring the liquid level to the reference mark.

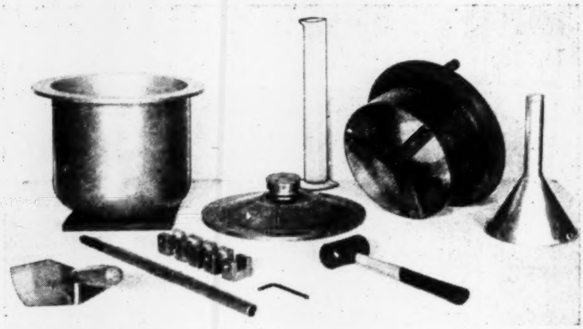

FIG. 3.—Equipment for Determining Entrained Air Content of Freshly-Mixed Concrete by the Modified Rolling Method.

When half filled, the 10 by 10-in. bowl in upper view accommodates a 0.22-cu. ft. sample of concrete.

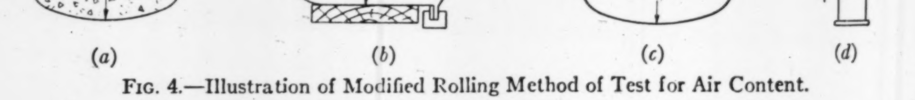

FIG. 4.—Illustration of Modified Rolling Method of Test for Air Content.

The entrained air is not easily removed from the concrete, and hence the rolling cycles (five revolutions in each inclined position) are repeated until an end point is reached, that is, until no more foam can be removed by rolling, rodding,[7] or other agitation, and therefore no more alcohol needs to be added to bring the liquid level to the reference mark.

The total volume of alcohol added is directly proportional to the volume of air removed from the 0.22 cu. ft. of concrete. It is not equal to it since there is a small correction for shrinkage of the alcohol-water mixture. With the apparatus shown, about 65 ml. of 91 or 99 per cent isopropyl alcohol is equivalent to 1 per cent air in the concrete. The graduate from which alcohol is poured and measured can be calibrated to indicate air content directly in per cent and tenths of a per cent.

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[6] Isopropyl alcohol has been found to be very effective in destroying the tough, finely divided foam which forms on the surface of the water when air is removed from the concrete. This alcohol is a definite chemical compound, is not denatured, and is not toxic but neither is it potable. It is widely available and relatively cheap.

[7] With nonworkable mixes of low slump, or with concrete samples previously subjected to the pressure test, it is desirable to rod the concrete thoroughly after the flask has been returned to its vertical position following the first rolling cycle. This is intended to break up compacted lumps of concrete.

Operation of Modified Rolling Method (Figs. 3 and 4):

This method employs a round-bottom flanged steel bowl nominally 10 in. in diameter and 10 in. deep having a volumetric capacity of about 0.44 cu. ft. However, in this method the bowl is only half filled with a 0.22-cu. ft. sample of concrete by the usual rodding procedure. This volume is obtained by the use of a metal strike-off device which functions as a tamper to get a smooth surface and as a gage to get the proper level. Concrete is added or removed from the bowl with a trowel until the proper volume is obtained. A conical cover provided with a neck, reference mark, and quick-closing cap is then clamped on the bowl against a rubber gasket making a watertight seal. Water is added until the water level reaches the reference mark on the inside of the neck. After sealing with the cap, the bowl is turned over on its side and rolled back and forth on a board or on suitable rollers. During rolling, the sides and bottom end of the bowl are vigorously tapped with a rawhide mallet. After rolling, the bowl is returned to an upright position and isopropyl alcohol added from the graduate to destroy the foam and bring the liquid to the reference mark.

After the amount of alcohol has been recorded, some of the liquid in the bowl is removed with a large syringe and carefully saved for later replacement in the bowl. A steel stirring rod ($\frac{5}{8}$ in. in diameter with a bullet-shaped end) is then inserted through the neck and the contents of the bowl thoroughly stirred. The liquid which was removed is replaced and more alcohol added if required.

The rolling cycles are repeated until no more foam appears and there is no apparent change in the liquid level. The total volume of alcohol added is directly proportional to the volume of air removed from the sample of concrete. With the 0.22-cu. ft. sample and the small correction for shrinkage of the alcohol-water mixture, 65 ml. of alcohol represent 1 per cent air in the concrete.

Appendix I gives information regarding the shrinkage of isopropyl alcohol-water mixtures and other considerations of interest in connection with the rolling methods of test.

PRESSURE METHOD

The pressure method is based on the principle for measuring air content proposed by Klein and Walker.[8] A sample of fresh concrete is placed in a closed pressure-tight vessel and air pressure is then applied to it to compress the entrained air. According to Boyle's law, the volume of a gas (at a given temperature) is inversely proportional to the pressure to which it is subjected, the volume of air in the concrete is determined by the amount it is compressed by a given increase in pressure.

Apparatus:

The general appearance of the pressure-type equipment developed by the Research Laboratories of the Portland Cement Assn., is shown in Fig. 5. The indicator and accessories are stored in a stout wood chest to facilitate transport. The chest also serves as a support for the indicator on the job and brings it to a convenient elevation for conducting the various operations of the test.

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[8] W. H. Klein and Stanton Walker, "A Method for Direct Measurement of Entrained Air in Concrete," *Journal*, Am. Concrete Inst., Vol. 17, No. 6, June, 1946, pp. 657-668; *Proceedings*, Am. Concrete Inst., Vol. 42, p. 657 (1946).

Operation of Pressure Method:

The operation of this apparatus will become clear from the diagrams of Fig. 6 and the remarks which follow. The round-bottom, flanged steel bowl, nominally 8 in. in diameter and 8 in. deep has a capacity of approximately 0.22 cu. ft. of concrete when struck off flush with the top of flange. After placing of the concrete, the cone-shaped cover is clamped to the flange of the bowl, a pressure-tight seal being obtained with a rubber gasket cemented to the flange of the cover. The space above the concrete is then filled with water to the zero mark on the graduated vertical precision bore glass tube attached to the apex of the cone (Fig. 6(*a*)). Pressure[9] is applied to the concrete by pumping air into the space over the water column until the pressure gage indicates the predetermined operating

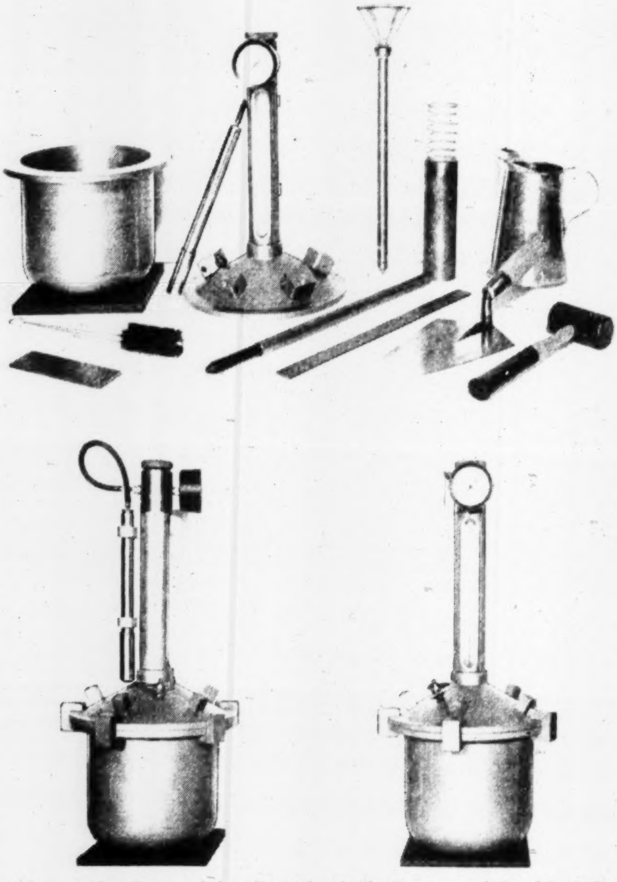

FIG. 5.—Equipment for Determining Entrained Air Content of Freshly-Mixed Concrete by the Pressure Method.

When filled, the 8 x 8-in. bowl in upper view accommodates a 0.22-cu. ft. sample of concrete. Lower views show side and front of the assembled pressure-type indicator.

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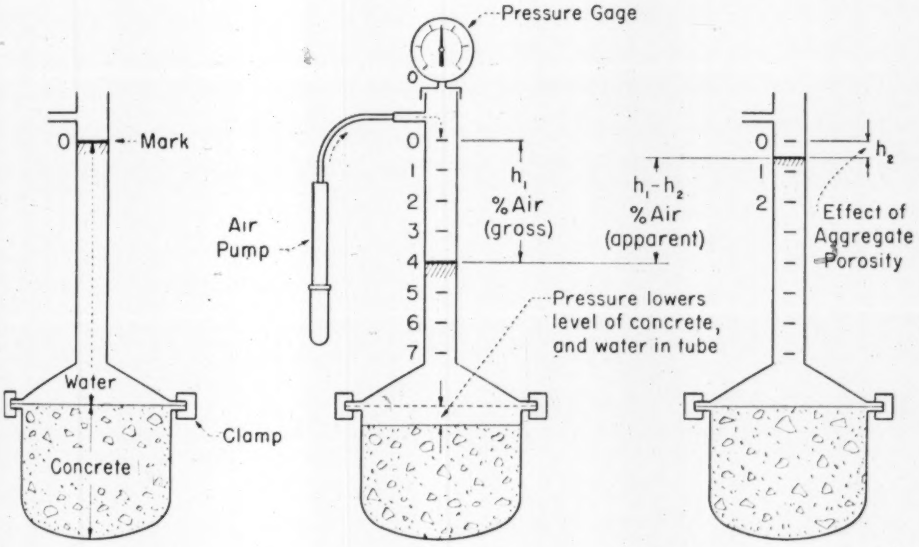
[9] The air pressure, which is furnished by a small hand pump, is indicated by a gage graduated in pounds and fifths of a pound, and having a range of 15 psi.

pressure, *P*.[10] The contraction in volume of the air in the concrete with increase in pressure is indicated by a lowering in level of the water in the graduated glass tube as shown in Fig. 6(*b*). The scale on this tube is graduated in inches and tenths of an inch but indicates the gross air content h_1 in per cent and tenths of a per cent when the proper operating pressure (determined by calibration against a known volume of air) is applied. When the pressure is released the level of the water column does not immediately return to the original zero mark but, because of penetration of water into the pores of the aggregate, remains at a level below the zero mark as indicated by the distance h_2 in Fig. 6(*c*). The ***apparent*** air content is the difference between the maximum reading h_1 and the minimum reading h_2. However, the *actual entrained air content* is not obtained until an aggregate correction factor is subtracted from the apparent air content to correct for the compression of the air in the aggregates when water is forced into the pores by the applied pressure. This correction factor varies from about 0.15 to 1.0 per cent of air for different natural aggregates used in concrete depending on the porosity and moisture condition of the aggregate. It is determined independently by applying the same test pressure that is applied to the concrete sample, to a sample of fine and coarse aggregate in approximately the same moisture condition, amount, and proportions as they occur in the concrete sample under test.



(*a*) (*b*) (*c*)

FIG. 6.—Illustration of Pressure Method of Test for Air Content.

Entrained air content = apparent air content minus the aggregate correction factor.

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[10] The operating pressure, *P*, will vary with the elevation above sea level and the characteristics of the pressure gage and the apparatus. It will be about 7.6 psi. at sea level (assuming a barometric pressure of 29.92 in. of mercury) and will decrease with decrease in barometric pressure to about 5.5 psi. at an elevation of 10,000 ft. above sea level (20.57 in. of mercury). However, extreme variations from average barometric pressure in a given locality of about 0.7 in. of mercury will alter the gage pressure by only about 0.15 psi. and can be ignored because it introduces an error of only about 0.05 percentage points of air when the entrained air content of the concrete is about 3.0 per cent.

Calibration of Indicator to Determine Proper Operating Pressure, P:

The accessories for the indicator shown in Fig. 5 include a cylindrical brass container of known volume and a coil spring. In the calibration of the indicator the cylinder is inverted within the bowl and held in place by the spring when the cover is clamped in position (Fig. 8 (*b*)). Small steel paper clips are used to hold the lower edge of the inverted cylinder about $\frac{1}{16}$ in. above the bottom of the bowl. When the assembled indicator is filled to the zero mark with water, the gage pressure, designated as P_c, is ascertained by pumping air into the assembly until the water level indicates the percentage of air that has been introduced by the inverted cylinder when the bowl is level full of water, plus an allowance of 0.05 per cent of air for expansion of the indicator under the applied pressure.[11] The proper gage pressure, P, to be used in determining the entrained air in concrete is, P_c minus 0.10 psi. The value 0.10 psi. is a correction for the difference in the distribution of air in a sample of fresh concrete and in the calibration cylinder. This item together with other theoretical and practical considerations underlying the pressure method is discussed in Appendix II.

The calibration procedure outlined eliminates the need for accurate pressure application in terms of pounds per square inch or other units so long as the needle of the pressure gage is brought to the same point determined in the calibration test. Any suspected change in the functioning of the gage needle (as might occur during shipping and handling of the equipment, etc.) can be checked by recalibrating to determine the proper operating pressure.

Checking Pressure Method by Rolling Method:

After completion of a test by the pressure method, the results may be checked by immediately testing the same concrete sample by the rolling method. With suitably designed apparatus this can be accomplished by clamping the covering bowl for the rolling method to the bowl containing the concrete sample used in the pressure test. The usual rolling method of test is then followed except that special precautions must be taken to loosen concrete which tends to be compacted at the bottom of the bowl as a result of the pressure application.

PART II.—SUGGESTED METHODS OF TEST FOR ENTRAINED AIR CONTENT OF FRESHLY MIXED CONCRETE BY THE ROLLING METHODS

Scope:

1. These methods of test cover the procedure for determining the entrained air content of freshly mixed concrete by the rolling and modified rolling methods.

NOTE.—These direct volumetric methods are believed to be adequate for all ordinary types of concrete including concrete made with highly porous aggregates (such as slag, etc.). With highly porous aggregates a correction factor (up to 0.5 per cent air) must be deducted from the indicated air content to compensate for the absorption of water that occurs during the test. This can be determined from separate tests on approximately the same amount of aggregate as is present in the concrete sample tested.

ROLLING METHOD

Apparatus and Materials:

2. The apparatus (see Fig. 1) and materials shall consist of the following:

(*a*) *Measuring Bowl.*—A flanged cylindrical steel bowl having a diameter equal to 1 to 1.25 times the height and a rounded bottom inside and outside. All exterior and interior surfaces of the bowl shall be smooth machined surfaces. The capacity of the bowl shall be not less than 0.20 cu. ft. for concrete containing aggregates of 2-in. maximum size and not less than 0.40 cu. ft. for concrete containing aggregates of 3-in. maximum size. The bowl shall be watertight and sufficiently rigid to withstand sharp and repeated blows from the rawhide mallet without permanent distortion. In general, this degree of rigidity will require a flange thickness of not less than $\frac{5}{16}$ in. and a bottom and side wall thickness of not less than $\frac{3}{16}$ in.

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[11] This allowance, called the expansion factor, corresponds to the additional amount the water column will lower because of the small increase in volume of the indicator which results from the application of internal pressure. To check the expansion factor for any pressure indicator, fill with water only, and apply the usual test pressure. The amount the water column lowers will be the equivalent expansion factor for that particular indicator.

(*b*) *Covering Bowl.*—This shall be of the same shape and dimensions as the measuring bowl except that it shall be fitted with a brass collar or neck about $1\frac{3}{4}$-in. in internal diameter and provided with a machined shoulder which serves as a reference mark for gaging the liquid level. A quick-closing cap shall provide a watertight seal over the opening in the neck. Rigid clamps shall be provided to make a watertight seal with the measuring bowl. The rubber gasket in the flange of the cover shall be designed to prevent entrapping air at the joint between the flanges of cover and bowl. The clamping arrangement shall avoid the use of notches or holes in the flanges of either bowl since they are likely to become filled with mortar.

(*c*) *Cradle.*—This device is used to tilt and roll the assembled flask (measuring and covering bowls) with its concrete and liquid contents in inclined positions. The cradle shall be light, rigid, and so designed that the flask may be easily rolled in two oppositely inclined positions in each of which the long axis of the flask makes an angle of about 30 deg. with the horizontal.

(*d*) *Trowel.*—A standard brick mason's trowel, nominally $5\frac{1}{4}$ in. in width and $10\frac{1}{2}$ in. in length, with the point cut off at right angles to the long axis of the blade at a distance of $8\frac{1}{4}$ in. from the heel of the trowel.

(*e*) *Tamping Rod.*—A straight steel tube $\frac{3}{4}$ in. in outside diameter, made from No. 14 gage material and 17 in. in length, threaded at one end for a hard rubber rod $\frac{5}{8}$ in. in diameter and protruding $3\frac{1}{2}$ in. The rubber insert shall be tapered for a distance of 1 in. to a spherically shaped end having a radius of approximately $\frac{1}{4}$ in.

(*f*) *Stirring Rod.*—A straight $\frac{5}{8}$ in. round metal rod, approximately 24 in. in length and tapered for a distance of 1 in. to a spherically shaped end having a radius of approximately $\frac{1}{4}$ in.

(*g*) *Rawhide Mallet.*—A standard size rawhide mallet weighing $7\frac{1}{2}$ oz., with a head $1\frac{3}{4}$ in. in diameter and $3\frac{1}{4}$ in. in length and a handle $8\frac{1}{2}$ in. in length.

(*h*) *Strike-Off Bar.*—A flat straight steel bar $\frac{1}{8}$ in. in thickness, 1 in. in width and 16 in. in length, with rounded edges as available in commercial hot-rolled flats.

(*i*) *Special Funnel.*—A funnel 6-in. in diameter at the top with a closed perforated spout. The spout shall consist of a brass tube about $1\frac{1}{4}$ in. in outside diameter and made from No. 16 gage material, provided with 16 evenly spaced holes made with a No. 35 drill $\frac{1}{4}$ in. from the closed end.

(*j*) *Measure for Water.*—A measure having a 1-gal. capacity.

(*k*) *Graduate.*—A cylindrical graduate of 500 ml. capacity graduated at intervals of 5 ml. and numbered one way with the zero mark at the top.

(*l*) *Syringe.*—A syringe having a spout 4 in. in length connected to a rubber bulb of at least 100 ml. capacity.

(*m*) *Thermometer.*—A rugged pocket-type thermometer 6 in. in length graduated from 0 to 220 F. in 2 deg. divisions.

(*n*) *Water.*—Any water suitable for use in concrete will be satisfactory for this test. The temperature of the water shall be within plus or minus 5 F. of the temperature of the mixed concrete.

(*o*) *Isopropyl Alcohol.*—A commercial grade of isopropyl alcohol with a minimum purity of 91 per cent alcohol by volume. Commercial grades designated as 91, 95, or 99 per cent isopropyl alcohol may be used interchangeably in the tests. The temperature of the alcohol shall be within plus or minus 5 F. of the temperature of the mixed concrete.

Calibration of Apparatus:

3. (*a*) The volume, *V*, of the measuring bowl shall be obtained by determining the weight of water at 70 F. (21.1 C.) required to fill the bowl, as follows:

A thin film of cup grease shall be smeared on the flange of the bowl. With a scale sensitive to 0.01 lb., the bowl shall be weighed together with a piece of plate glass sufficiently large to cover its open end. The bowl shall then be filled with water so that the level is slightly above the rim. The glass plate shall be slid carefully over the top edge of the bowl, using a small rubber syringe to remove excess water as required to keep the bowl from overflowing. Care shall be taken to avoid trapping air under the plate. Any water on the outside of the bowl and glass plate shall be wiped off and the bowl weighed, together with the water and plate. The difference between the first and second weighings gives the water content, *W*, of the bowl in pounds, which divided by 62.31 gives the volume, *V*, in cubic feet.

(*b*) With a sturdily constructed measuring bowl and cover, the calibration for volume need be made only once (at the time of initial calibration), or only occasionally as a check on the volume of concrete that is determined by the measuring bowl and strike-off bar (Note).

NOTE.—Although it is desirable to obtain uniformity in the volume of the concrete samples tested, it should be pointed out that the degree of precision in volume required in the gravimetric method[3] is not necessary in either these rolling methods or the pressure method.[12] For example, with a bowl 8 in. in depth, a difference in level of $\frac{1}{8}$ in. above or below the contemplated level will affect the volume by only about 1 per cent (1 part in 96) and will introduce an error in the percentage of entrained air of only about 0.03 when the entrained air content of the concrete is about 3.0 per cent.

Determination of Aggregate Correction Factor for Highly Porous Aggregates:

4. (*a*) With highly porous aggregates, a significant amount of water may enter the pores during the rolling process and hence a greater volume of liquid (alcohol) will have to be added to restore the original level of water over the concrete to the reference mark in the neck of the flask. As a result, the indicated amount of air is higher than the entrained air actually present in the concrete. In such a case, the indicated air content shall be corrected by deducting a value called the aggregate correction factor. This factor shall be determined independently by observing the amount of liquid that must be added in a separate test on a combined sample of fine and coarse aggregate in approximately the same moisture condition, amount, and proportions as they occur in the concrete sample under test, as described in Paragraphs (*b*) to (*e*).

(*b*) The weights of fine and coarse aggregate that would normally be present in the volume, *V*, of a sample of fresh concrete whose entrained air content is to be determined shall be calculated as follows:

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[12] Suggested Method of Test for Entrained Air Content of Freshly Mixed Concrete by the Pressure Method, see p. 847.

$$w_f = \frac{V}{S} \times W_f \quad \text{..........(1)}$$

$$w_c = \frac{V}{S} \times W_c \quad \text{..........(2)}$$

where:

w_f = weight of fine aggregate in concrete sample under test, in pounds,

V = volume of concrete sample (same as volume of measuring bowl), in cubic feet,

S = volume of concrete produced per batch (Note), in cubic feet,

W_f = total weight of fine aggregate in batch in condition used, in pounds,

w_c = weight of coarse aggregate in concrete sample under test, in pounds, and

W_c = total weight of coarse aggregate in batch in condition used, in pounds.

NOTE.—The volume of concrete produced per batch can be determined in accordance with Section 6 (*b*) of the Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (A.S.T.M. Designation: C 138).[3]

(*c*) Representative samples of fine aggregate, of weight w_f, and of coarse aggregate, of weight w_c, shall be inundated in water in separate containers and allowed to absorb water for 5 min. to bring each sample to a moisture content comparable to that which obtains at the time the concrete is sampled. After draining off excess water, the aggregate shall be promptly placed in the bowl, filled one-third full of water, by adding alternately one scoop of fine aggregate and two scoops of coarse aggregate until all of the aggregate is inundated. Each scoopful shall be added in a manner that will entrap as little air as possible, and accumulations of foam shall be removed promptly. The sides of the bowl shall be tapped and the upper inch of the aggregate lightly rodded about ten times after each addition of sand to eliminate entrapped air.

(*d*) When all of the aggregate has been placed in the bowl, all foam and excess water shall be struck off and the flanges of both the bowl and cover shall be cleaned thoroughly so that when the cover is clamped in place a watertight seal will be obtained. The test shall be completed as described in Section 5(*b*) to (*d*).

(*e*) The aggregate correction factor, A_2, expressed as a percentage by volume of concrete, shall be calculated as follows:

$$A_2 = 0.00339 \frac{V_b}{V} \quad \text{..........(1)}$$

where:

V_b = total volume of alcohol (in milliliters) used to restore the liquid level in the test on the aggregate, and

V = volume of concrete sample (in cubic feet) used for the test described in Section 5.

Procedure for Determining Entrained Air Content of Concrete:

5. (*a*) A representative sample of the concrete to be tested shall be placed in the calibrated measuring bowl in three equal layers, each consolidated by rodding (or vibration) and by tapping the bowl. When the concrete is to be placed by rodding, each layer of concrete shall be consolidated by about 25 strokes of the tamping rod evenly distributed over the cross-section. The rodding of each layer shall be followed by tapping the sides of the bowl smartly 10 to 15 times with the rawhide mallet until the cavities left by rodding are leveled out and no large bubbles of air appear on

the surface of the rodded layer. In rodding the first layer, the rod shall not forcibly strike the bottom of the bowl. In rodding the second and final layers, only enough force shall be used to cause the rod to penetrate the surface of the previous layer. The bowl shall be just perceptibly overfilled by the third layer and the excess removed by sliding the strike-off bar across the top flange with a sawing motion until the bowl is just level full. Vibration may be substituted for rodding the sample when the air content of concrete placed by vibration is to be determined.

(*b*) The flanges of the bowl and of the cover shall be cleaned thoroughly so that when the cover is clamped in place a watertight seal will be obtained. The assembled flask then shall be carefully brought to a vertical position and water (Note) added over the concrete (without eroding or agitating the concrete) until it rises to the zero mark in the neck of the cover (see Fig. 2 (*a*)). During filling, the sides and top of the cover shall be lightly tapped with the mallet to remove any entrapped air bubbles above the concrete sample. Any foam on the surface of the water in the neck shall be removed with a spray of alcohol so that the water can be brought exactly to the zero mark before the cap is fastened in place.

NOTE.—The temperature of the water and alcohol used in the test shall be within plus or minus 5 F. of the temperature of the mixed concrete.

(*c*) The flask shall be inclined about 30 deg. from horizontal by means of the cradle so that the concrete can be tumbled from the upper to the lower end of the flask during rolling. The flask shall be slowly rotated on the rollers of the cradle for about ten revolutions while the upper end and sides are tapped sharply with the rawhide mallet to loosen the concrete and entrapped air. The flask shall then be inclined in the opposite direction by means of the cradle and again slowly rotated for about ten revolutions while tapping with the mallet. The flask shall then be returned to its original upright position, the sides vigorously tapped, and isopropyl alcohol then added from the graduate to destroy as much of the accumulated foam as possible and to bring the liquid level to the zero mark. The amount of alcohol added shall be recorded. Some of the liquid shall then be removed from the flask with the syringe and carefully saved for later replacement in the bowl, and the concrete shall be thoroughly rodded to break up any lumps and to loosen concrete adhering to the bottom of the bowl. The liquid that was removed shall be returned to the flask, more alcohol added as needed to bring the liquid level to the zero mark, and the total amount of alcohol added shall be recorded.

(*d*) The rolling cycles (five revolutions in each inclined position) shall be repeated until an end point is reached; that is, until no more foam can be removed by rolling, tapping, rodding, or other agitation, and therefore no more alcohol needs to be added to bring the liquid level to the zero mark. The end point shall be considered as reached when two successive readings of the volume of alcohol used agree within 0.05 per cent of air content. The final volume of alcohol, V_b, in milliliters, shall be used to calculate the entrained air content, A, in accordance with Section 6.

Calculation:

6. The entrained air content of the concrete shall be calculated as follows (Note):

$$A = 0.00339 \frac{V_b}{V} - A_2 \quad \ldots\ldots \quad (1)$$

where:

A = entrained air content, percentage by volume of concrete,

V_b = total volume of isopropyl alcohol used to restore liquid level, in milliliters,

V = volume of concrete sample, in cubic feet,

A_2 = aggregate correction factor, percentage by volume of concrete, determined in accordance with Section 4. For most natural aggregates the value of A_2 can be assumed to be negligible.

NOTE.—The value for A given in Eq. 1 is derived from the more general expression,

$$A = \frac{0.0000353 V_b}{V} \times C \times R \times 100 - A_2 \ldots (2)$$

where:

C = compression correction factor (discussed in Appendix I, which is equal to 1.022 at sea level, 1.026 at 5000 ft. above sea level, and 1.035 at 13,000 ft. above sea level, and

R = alcohol contraction factor (discussed in Appendix I).

Most of the tests for entrained air content will probably be made between sea level and an elevation of 5000 ft. on concrete having an air content between 2 and 8 per cent and a temperature between 68 and 86 F. after mixing. It is contemplated that isopropyl alcohol of either 91 or 99 per cent strength will be available. No significant error will be introduced in the value of the entrained air content, A, for this range of conditions if the value C is taken as 1.024 and the value of R as 0.938. For these conditions,

$$A = 0.00339 \frac{V_b}{V} - A_2 \ldots\ldots\ldots (1)$$

For most natural aggregates the correction factor, A_2, can be assumed to be negligible. When the value for V is inserted in Eq. 1, the entrained air content becomes a simple function of the volume, V_b, of isopropyl alcohol used.

MODIFIED ROLLING METHOD

Apparatus and Materials:

7. The apparatus (see Fig. 3) and materials shall be as described in Section 2 for the rolling method, with the following exceptions:

(*a*) *Measuring Bowl.*—A flanged cylindrical steel bowl having a diameter equal to its height, with rounded bottom inside and outside. All exterior and interior surfaces of the bowl shall be smooth machined surfaces. The internal volume of the bowl shall be such that when half filled the bowl will accommodate not less than a 0.20-cu. ft. sample of concrete for concrete containing aggregates of 2- in. maximum size and not less than a 0.40 cu. ft. sample of concrete for concrete containing aggregates of 3 in. maximum size. The bowl shall be watertight and sufficiently rigid to withstand sharp and repeated blows from the rawhide mallet without permanent distortion. In general, this degree of rigidity will require a flange thickness of at least $\frac{5}{16}$ in. and a bottom and sidewall thickness of at least $\frac{3}{16}$ in.

(*b*) *Conical Cover*—A flanged conical steel or iron cover provided with a neck, reference mark, quick-closing cap, and clamps as described for the covering bowl used in the Rolling Method. (Section 2(*b*)). All exterior and interior surfaces of the cover shall be smooth machined surfaces. The sloping interior surface shall be inclined at an angle of not less than 30 deg. with the horizontal.

(*c*) *Cradle.*—The flask formed by the assembled measuring bowl and its cover may be rolled either on a board or on suitable rollers. Preferably, a cradle, similar to the device described for the rolling method, should be available to permit rolling in inclined positions.

(*d*) *Strike-Off Gage.*—This device functions as a tamper to get a smooth surface and as a gage to get the proper level of concrete in the measuring bowl. These functions can be performed by several designs, one of which is shown in Fig. 3.

Calibration of Apparatus:

8. (*a*) The volume, V, of the bowl at the level of strike-off gage shall be obtained by determining the weight of water at 70 F. (21.1 C.) required to fill the bowl so that the water just makes contact with the lower edges of the strike-off gage. This gage should rest on the flange of the bowl in the same manner as when it is used to gage the level of the concrete sample in Section 10(*a*). The water content, W, of the bowl in pounds, divided by 62.31 gives the volume, V, in cubic feet and represents the volume of the concrete sample determined by the strike-off gage.

(*b*) With a sturdily constructed measuring bowl and cover, the calibration for volume need be made only once (at the time of initial calibration), or only occasionally as a check on the volume of concrete that is determined by the measuring bowl and strike-off gage (Note, Section 3).

Determination of Aggregate Correction Factor for Highly Porous Aggregates:

9. Determine the aggregate correction factor for highly porous aggregates in accordance with Section 4, except that the test shall be completed in accordance with Section 10(*b*) to (*d*) instead of Section 5(*b*) to (*d*).

Procedure for Determining Entrained Air Content of Concrete:

10. (*a*) A representative sample of concrete shall be placed in the measuring bowl as described in Section 5(*a*), except that the bowl shall be only partially filled with concrete (see Fig. 4) and leveled by means of the special strike-off gage (Section 7(*d*)) instead of the strike-off bar.

(*b*) The bowl and conical cover shall be assembled and filled with water (Note) as described in Section 5(*b*).

NOTE.—The temperature of the water and alcohol used in the test shall be within plus or minus 5 F. of the temperature of the mixed concrete.

(*c*) After closing the flask with the cap, it shall be turned over on its side and slowly rolled back and forth either on a board or on rollers. The end and sides of the flask shall be sharply tapped with the mallet during rolling. After about 20 revolutions, the flask shall be returned to its original upright position and isopropyl alcohol (Note) added from the graduate to destroy as much of the foam as possible and to bring the liquid level to the zero mark. The amount of alcohol added shall be recorded. Some of the liquid shall then be removed from the flask with the syringe and carefully saved, and the concrete shall then be thoroughly rodded and stirred. The liquid that was removed shall be returned to the flask, more alcohol added as needed to bring the liquid level to the zero mark, and the total amount of alcohol added shall be recorded.

(*d*) The rolling cycles (10 revolutions per cycle) shall be repeated until no more foam appears and there is no apparent change in the liquid level. The end point shall be considered as reached when two successive readings of the volume of alcohol used agree within 0.05 percent of air content. The final volume of alcohol, V_b, in milliliters shall be used to calculate the entrained air content, A, in accordance with Section 11.

Calculation:

11. Calculate the entrained air content of the concrete as described in Section 6, using for the aggregate correction factor, A_2, the value determined in accordance with Section 9.

PART III.—SUGGESTED METHOD OF TEST FOR ENTRAINED AIR CONTENT OF FRESHLY MIXED CONCRETE BY THE PRESSURE METHOD

Scope:

1. This method of test covers the procedure for determining the entrained air content of freshly-mixed concrete by the pressure method.

NOTE.—This method is believed to be adequate for all ordinary types of concrete and mortar, except for concretes or mortars made with highly porous aggregates (such as slag, etc.) where the aggregate correction factor cannot be determined accurately by the technique found satisfactory for the usual types of relatively dense natural aggregates.

Apparatus:

2. The apparatus (see Fig. 5) shall consist of the following:

(*a*) *Measuring Bowl.*—A flanged cylindrical steel bowl having a diameter equal to 1 to 1.25 times the height and a rounded bottom inside and outside. The outer rim and upper surface of the flange, as well as the interior surfaces of the bowl, shall be smooth machined surfaces. The capacity of the bowl shall be not less than 0.20 cu. ft. for concrete containing aggregates of 2-in. maximum size and not less than 0.40 cu. ft. for concrete containing aggregates of 3-in. maximum size. The bowl shall be pressuretight and sufficiently rigid to limit the expansion factor of the indicator assembly to not more than 0.10 per cent of air content on the indicator scale when under the normal operating pressure.

(*b*) *Conical Cover Assembly.*—A flanged cover, preferably of steel or hard metal having a high modulus of elasticity, and not readily attacked by the cement paste, with interior surfaces inclined not less than 30 deg. from the horizontal. The outer rim and lower surface of the flange and the sloping interior surface shall be smooth machined surfaces. The cover shall be pressuretight and sufficiently rigid to limit the expansion factor of the indicator assembly as prescribed in Paragraph (*a*).

The cover shall be fitted with a vertical tube of uniform cross-section accurately graduated to indicate the changes in water level which correspond to the air content of the concrete. This tube shall be a graduated precision-bore glass tube with graduations etched in inches and tenths of inches (for a distance of 8 in.) to represent air content in per cent and tenths of a per cent when the proper gage pressure determined by the calibration test is applied. The internal diameter of the tube shall be designed so that under a normal operating pressure of 7.5 ± 0.5 psi. the water column will lower approximately 1 in. for each per cent of air introduced by the calibration cylinder. The applied air pressure shall be indicated by a pressure gage connected to the air chamber above the water column. The gage shall have a range of 15 psi. with major graduations in pounds per square inch (identified by individual numerals) and minor graduations in 0.20 psi. The gage shall have a dial at least $2\frac{1}{2}$-in. in diameter and be provided with a center-back connection for rigidity.

The cover shall be fitted with a suitable vent (preferably a screwed cap) at the top of the air chamber, a small hand-operated air pump, an air valve, and a petcock for bleeding off water as required. Suitable means for clamping the cover to the bowl shall be provided to make a pressuretight seal without entrapping air at the joint between the flanges of the cover and bowl. The clamping arrangement shall avoid notches or holes in the flanges that are likely to be filled with mortar.

(*c*) *Calibration Cylinder.*—A cylindrical measure having an internal volume equal to approximately 6.5 per cent of the volume of the measuring bowl and an inside depth $\frac{1}{2}$ in. less than that of the bowl. A satisfactory measure may be machined from No. 16 gage brass tubing (of proper diameter to provide the volume desired) to which a brass disk $\frac{1}{4}$ in. in thickness is soldered to form the bottom.

(*d*) *Coil Spring.*—A compression spring, made of $\frac{1}{8}$-in. round steel wire, 2 in. in outside diameter and $1\frac{3}{4}$ in. in inside diameter to hold the calibration cylinder firmly in position during the calibration test.

(*e*) *Trowel.*—A standard brick mason's trowel, nominally $5\frac{1}{4}$ in. in width and $10\frac{1}{2}$ in. in length, with the point cut off at right angles to the long axis of the blade at a distance of $8\frac{1}{4}$ in. from the heel of the trowel.

(*f*) *Tamping Rod.*—A straight steel tube $\frac{3}{4}$ in. in outside diameter, made from No. 14 gage material, and 17-in. in length, threaded at one end for a hard rubber rod $\frac{5}{8}$ in. in diameter and protruding $3\frac{1}{2}$ in. The rubber insert shall be tapered for a distance of 1 in. to a spherically shaped end having a radius of $\frac{1}{4}$ in.

(*g*) *Rawhide Mallet.*—A standard size rawhide mallet, weighting $7\frac{1}{2}$ oz., with a head $1\frac{3}{4}$ in. in diameter and $3\frac{1}{4}$-in. in length and a handle $8\frac{1}{2}$ in. in length.

(*h*) *Strike-Off Bar.*—A flat straight steel bar $\frac{1}{8}$ in. in thickness, 1 in. in width, and 16 in. in length, with rounded edges as available in commercial hot-rolled flats.

(*i*) *Spray Tube.*—A brass tube $\frac{3}{4}$ in. in outside diameter and made of No. 18 gage material, provided with a closed perforated end (12 evenly spaced holes made with No. 47 drill) for filling the indicator with water without eroding or agitating the concrete. The tube shall be designed so that when it is inserted for its full length into the vertical tube of the conical cover any water added at the open end is sprayed horizontally from the perforated end to the walls of the conical cover.

(*j*) *Funnel* with spout fitting into spray tube.

(*k*) *Measure for Water.*—A measure having a $\frac{1}{2}$- or 1-gal. capacity, as required, to fill the indicator with water from the top of the concrete to the zero mark.

(*l*) *Tube Brush* for cleaning the inside of the graduated tube.

Calibration of Apparatus:

3. Change in barometric pressure with change in elevation and rough handling under job conditions will affect the calibration of the pressure-type indicator. The steps described in Paragraphs (*a*) to (*e*) inclusive, are prerequisites for the final calibration test to determine the operating pressure, *P*, on pressure gage as described in Paragraph (*f*). Normally the steps in Paragraphs (*a*) to (*e*) need only be made once (at the time of the initial calibration), or only occasionally to check volume constancy of the calibration cylinder and measuring bowl. On the other hand, the calibration test described in Paragraph (*f*) must be made as frequently as necessary to insure that the proper gage pressure, *P*, is being used in tests for the entrained air content of concrete. Moreover, a change in elevation of more than 300 ft. from the location at which the indicator was last calibrated will require recalibration in accordance with Paragraph (*f*).

(*a*) *Volume, v, of Calibration Cylinder.*—The volume, *v*, of the calibration cylinder shall be obtained by determining the weight of water at 70 F. (21.1 C.) required to fill it, as follows: Using a scale sensitive to 0.5 g. the cylinder shall be weighed together with a piece of plate glass sufficiently large to cover its open end. The cylinder shall then be filled with water so that the level is slightly above the rim. The glass plate shall be slid carefully over the top edge of the cylinder, using a small rubber syringe to remove excess water as required to keep the cylinder from overflowing. Care shall be taken to avoid trapping air under the plate. Any water on the outside of the container and glass plate shall be wiped off and the container weighed, together with the water and plate. The difference between the first and second weighings gives the water content, w, of the cylinder in grams, which divided by 0.998 gives the volume, v, in milliliters.

(*b*) *Volume, V, of Measuring Bowl of Indicator.*—The volume, V, of the measuring bowl of the indicator shall be obtained by determining the weight of water at 70 F. (21.1 C) required to fill it. The procedure described in Paragraph (*a*) shall be followed, using a scale sensitive to 0.01 lb. A thin film of cup grease smeared on the flange of the bowl will make a watertight joint between the glass plate and top of bowl. The water content, W, of the bowl in pounds, divided by 62.31 gives the volume, V, in cubic feet (Note 1).

NOTE. 1—Although it is desirable to obtain uniformity in the volume of the concrete samples tested, it should be pointed out that the degree of precision in volume required in the gravimetric method[3] is not necessary in either the pressure method or the rolling methods.[13] For example, with a bowl 8 in. in depth a difference in level of $\frac{1}{8}$ in. above or below the contemplated level will affect the volume by only about 1 per cent (1 part in 96) and will introduce an error in the percentage of entrained air of only about 0.03 when the entrained air content of the concrete is about 3.0 per cent.

(*c*) *Determination of Constant R.*—The constant R represents the volume of the calibration cylinder expressed as a percentage of the volume of the indicator bowl and shall be calculated by either of the following formulas:

$$R = 0.003531 \frac{v \text{ (in milliliters)}}{V \text{ (in cubic feet)}} \text{ (volumetric)} \quad \ldots (1)$$

$$R = 0.2205 \frac{w \text{ (in grams)}}{W \text{ (in pounds)}} \quad \text{(weight)} \quad \ldots\ldots (2)$$

(*d*) *Determination of Expansion Factor, D.*—The expansion factor, D, (Note 2) for any given pressure indicator shall be determined by filling it with water only (making certain that all entrapped air has been removed and the water level is exactly on the zero mark) and applying an air pressure approximately equal to the operating pressure, P, determined by the calibration test described in Paragraph (*f*). The amount the water column lowers will be the equivalent expansion factor D for that particular indicator and pressure (Note 3).

NOTE. 2—Although the bowl, cover and clamping mechanism of a pressure-type indicator must of necessity be sturdily constructed so that it will be reasonably pressuretight, the application of internal pressure will result in a small expansion in volume. This expansion will not effect the test results because, with the procedure described in Sections 5 and 6, the amount of expansion is the same for the test for entrained air in concrete as for the test for aggregate correction factor on combined fine and coarse aggregates, and is thereby automatically cancelled. However, it does enter into the calibration test to determine the air pressure to be used in testing fresh concrete and appears as the value D in the expression for the calibration factor, K, in Eq. (3) of Paragraph (*e*). The effect of expansion must also be considered when the aggregate correction factor is determined from tests on separate samples of fine and coarse aggregate as described in Appendix III.

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[13] Suggested Method of Test for Entrained Air Content of Freshly Mixed Concrete by the Rolling Methods, see p. 840.

NOTE 3.—It will be sufficiently accurate for this purpose to use an approximate value for P determined by making a preliminary calibration test as described in paragraph *(f)*, except that an approximate value for the calibration factor, K, shall be used. For this test $K = 0.98R$ which is the same as Eq. 3 of Paragraph *(e)* except that the expansion factor D, as yet unknown, is assumed to be zero.

(*e*) *Determination of Calibration Factor K*.—The calibration factor, K, is the amount the water column must be depressed during the calibration procedure to obtain the (gage) pressure required to make the graduations on the glass tube correspond directly to the percentage of air introduced into the indicator bowl by the calibration cylinder when the bowl is level full of water. The value K shall be calculated as follows (Note 4):

$$K = 0.98R + D \qquad (3)$$

where D = expansion factor of indicator as determined in Paragraph (*d*).

NOTE 4.—The value for K given in Eq. 3 is derived from the more general expression

$$K = \frac{v}{V} \times 100 \times H + D \qquad (4)$$

where H = ratio of the volume of air in the calibration cylinder after inundation to the volume before inundation under a head of $r - t_1$ inches of water (see Eqs. 18 and 21, Case 2, Appendix II). H decreases slightly as the elevation above sea level increases and is about 0.980 at sea level, 0.975 at 5000 ft. above sea level, and 0.970 at 13,000 ft. above sea level. The error introduced by neglecting these variations in the value of H will usually be so small (corresponding to less than 0.05 per cent air) that Eq. 3, $K = 0.98R + D$, usually will be sufficiently accurate. However, the value of H should be checked for each design of indicator from Eq. 21,

$H = \frac{m - t_1}{m}$ where t_1 is obtained from Eq. 18 which is $t_1^2 - (B + r + m)\, t_1 + mr = 0$, as given in Case 2 of Appendix IV.

(*f*) *Calibration Test to Determine Operating Pressure, P, on Pressure Gage.*—The rim of the calibration cylinder shall be fitted with three steel paper clips equally spaced around the circumference before the cylinder is inverted and placed at the center of the dry bottom of the indicator bowl. These clips provide an opening for flow of water into the calibration cylinder when pressure is applied. The compression spring shall then be placed on top of the inverted cylinder and the conical cover carefully lowered to maintain good vertical alignment as it makes contact with the spring and the flange of the bowl.

After the cover is clamped in place the indicator shall be carefully adjusted to a vertical position and water added by means of the spray tube and funnel until it rises above the zero mark (See Fig. 6) The vent shall then be closed and air pumped into the indicator to a pressure of about 8 psi. The assembly shall be inclined about 30 deg. from vertical (Note 5) and, using the bottom of the bowl as a pivot, several complete circles shall be described with the upper end of the column, simultaneously tapping the cover and sides of bowl lightly to remove any entrapped air adhering to the inner surfaces of the apparatus.

The indicator shall then be returned to a vertical position, the air pressure *gradually* released (to avoid loss of air from the calibration cylinder), and the vent opened. The water level then shall be brought exactly to the zero mark by bleeding water through the petcock in the top of the conical cover.

After closing the vent, pressure shall be applied until the water level has dropped an amount equivalent to about 0.1 to 0.2 per cent of air more than the value of the calibration factor, K, determined as described in Paragraph (*e*). To relieve local restraints, the sides of the bowl shall be tapped lightly, and, when the water level is exactly at the value of factor K, the pressure P_c indicated by the gage shall be read and recorded to the nearest 0.10 psi. The pressure shall then be ***gradually*** released and the vent opened to determine whether the water level returns to the zero mark when the sides of the bowl are tapped lightly (failure to do so indicates loss of air from the calibration cylinder or loss of water due to a leak in the assembly). If the water level fails to return to within 0.05 per cent air of the zero mark and no leakage beyond a few drops of water is found, some air probably was lost from the calibration cylinder. In this case, the calibration procedure should be repeated step by step from the beginning of this paragraph. If the leakage is more than a few drops of water, the leaking joint should be tightened before the calibration procedure is repeated. The indicated pressure reading shall be checked promptly by first bringing the water level exactly to the zero mark, closing the vent, and applying about 0.10 psi. (Note 6) more than the pressure P_c just determined. The gage shall be tapped lightly with a finger, and when it indicates the exact pressure P_c the water column should read the value of the calibration factor K used in the first pressure application within about 0.05 per cent of air.

The operating pressure, P, on the pressure gage for testing the entrained air in fresh concrete shall be the gage pressure, P_c, minus 0.10 psi. That is,

$$P = P_c - 0.10 \qquad (5)$$

NOTE 5: ***Caution:*** Indicator must not be moved from the vertical position until a pressure of 8 psi. has been applied which will force water about one third of the way up into the calibration cylinder. Any loss of air from this cylinder will nullify the calibration.

NOTE 6.—The value 0.10 psi. is the approximate value of a correction introduced by the difference between the distribution of air in a sample of fresh concrete and in the calibration cylinder. It is a theoretical correction which is described in Appendix II and is based on the approximate difference in pressure, P, given by Eq. 16 for Case 1 *versus* Eq. 16 for Case 2, for the 0.22 cu. ft. indicator shown in Fig. 5. A similar comparison for an indicator of twice this capacity (0.44 cu. ft.) shows that this correction will be about 0.07 psi. Although this correction and its effect is small for either size of indicator and probably could be neglected, it is included in Eq. 5 for the operating pressure, P, until practical considerations and experience with different pressure-type indicators show that it can be omitted.

Determination of Aggregate Correction Factor:

4. (*a*) Most concrete aggregates are more or less porous, and during application of pressure to the concrete sample as described in Section 5, water is forced into the pores and the air in the pores is compressed. The apparent air content must therefore be corrected by deducting a value depending on the response of the aggregate to the same (gage) pressure, P, that is applied in the test of the fresh concrete. This value is called the aggregate correction factor and can be determined independently (except for highly porous aggregates) by applying the desired pressure to a sample of inundated fine and coarse aggregate in approximately the same moisture condition, amount, and proportions as they occur in the concrete sample under test. In determining this factor it is important to follow the same sequence of pressure application and release as in the tests with concrete in order to reduce the effects of variations in time because the degree of penetration of moisture into the pores of the aggregate is a time function. The aggregate correction factor shall be determined on a combined sample of fine and coarse aggregate as directed in Paragraphs (*b*) to (*d*).

(*b*) The weight of the fine aggregate and of the coarse aggregate present in the volume, V, of a sample of fresh concrete whose entrained air content is to be determined shall be calculated as follows:

$$w_f = \frac{V}{S} \times W_f \dots\dots\dots\dots (1)$$

$$w_c = \frac{V}{S} \times W_c \dots\dots\dots\dots (2)$$

where:

w_f = weight of fine aggregate in concrete sample under test, in pounds,

V = volume of concrete sample (same as volume of measuring bowl), in cubic feet,

S = volume of concrete produced per batch (Note 1), in cubic feet,

W_f = total weight of fine aggregate in batch in condition used, in pounds,

w_c = weight of coarse aggregate in concrete sample under test, in pounds, and

W_c = total weight of coarse aggregate in batch in condition used, in pounds.

NOTE 1.—The volume of concrete produced per batch can be determined in accordance with Section 6 (*b*) of the Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (A.S.T.M. Designation: C 138).[3]

(*c*) Representative samples of fine aggregate, of weight w_f, and of coarse aggregate, of weight w_c, shall be inundated in water in separate containers and allowed to absorb water for 5 min. to bring each sample to a moisture content comparable to that which is obtained at the time the concrete is sampled. After draining off excess water, the aggregate shall be promptly placed in the bowl, filled one-third full of water, by adding alternately one scoop of fine aggregate and two scoops of coarse aggregate until all of the aggregate is inundated. Each scoopful shall be added in a manner that will entrap as little air as possible and accumulations of foam shall be removed promptly. The sides of the bowl shall be tapped and the upper inch of the aggregate lightly rodded about ten times after each addition of sand to eliminate entrapped air.

(*d*) When all of the aggregate has been placed in the bowl all foam and excess water shall be struck off and the flanges of both the bowl and conical cover shall be cleaned thoroughly so that when the cover is clamped in place a pressuretight seal will be obtained. The test shall be completed as described in Section 5 (*b*) and (*c*). The aggregate correction factor, A_2, is equal to $h_1 - h_2$ *as determined in the tests on the aggregate.*

NOTE 2.—The aggregate correction factor will vary with different aggregates. It can only be determined by test, since apparently it is not directly related to absorption of the particles. The test can be easily made and must not be ignored. Ordinarily the factor will remain reasonably constant for given aggregates, but an occasional check test is recommended. Appendix III gives some comments on tests for the aggregate correction factor and includes a procedure for determining this factor from tests on *separate samples* of fine and coarse aggregate.

Procedure for Determining Entrained Air Content of Concrete:

5.(*a*) A representative sample of the concrete to be tested shall be placed in the measuring bowl in three equal layers, each consolidated by rodding (or vibration) and by tapping the bowl. When the concrete is to be placed by rodding, each layer of concrete shall be consolidated by about 25 strokes of the tamping rod evenly distributed over the cross-section. The rodding of each layer shall be followed by tapping the sides of the bowl smartly 10 to 15 times with the rawhide mallet until the cavities left by rodding are leveled out and no large bubbles of air appear on the surface of the rodded layer. In rodding the first layer, the rod shall not forcibly strike the bottom of the bowl. In rodding the second and final layers, only enough force shall be used to cause the rod to penetrate the surface of the previous layer. The bowl shall be slightly overfilled by the third layer and the excess removed by sliding the strike-off bar across the top flange with a sawing motion until the bowl is just level full. Vibration may be substituted for rodding the sample when the air content of concrete placed by vibration is to be determined.

(*b*) The flange of the bowl and that of the conical cover shall be cleaned thoroughly so that when the cover is clamped in place a pressuretight seal will be obtained. The apparatus shall be assembled (see Fig. 6) and water then added over the concrete (without eroding or agitating the concrete) until it rises to about the halfway mark in graduated tube of the indicator. The assembly shall be inclined about 30 deg. from vertical and, using the bottom of the bowl as a pivot, several complete circles shall be described with the upper end of the column, simultaneously tapping the conical cover lightly to remove any entrapped air bubbles above the concrete sample. The indicator assembly shall be returned to its vertical position and the water column filled slightly above the zero mark, while lightly tapping the sides of the bowl. Any foam on the surface of the water column shall be removed with a syringe or with a spray of alcohol until a clear meniscus is obtained. The water level shall then be brought exactly to the zero mark of graduated tube before closing the vent at the top of the water column.

(*c*) Slightly more than the desired test pressure, P, (about 0.20 psi. more) shall be applied to the concrete by means of the small hand pump. To relieve local restraints, the sides of the measure shall be tapped lightly and, when the pressure gage indicates the exact test pressure, P, (as determined in accordance with Section 3 (*f*) in the calibration test) the water level, h_1, shall be read and recorded to the nearest division or half division (0.10 or 0.05 per cent air content) on the graduated tube of the water column (for example: 3.1, 4.05, 3.6, 3.85, etc.). The air pressure shall then be gradually released through the vent at the top of the water column and the sides of the bowl tapped lightly for about 1 min., after which the water level, h_2, shall be recorded to the nearest division or half division. The apparent air content, A_1, is equal to $h_1 - h_2$ (Fig. 6).

(*d*) The steps described in the Paragraph (*c*) shall be repeated (without adding water to reestablish the water level at the zero mark). The two consecutive determinations of apparent air content should check within 0.2 of a per cent of air and shall be averaged to give the value A_1 to be used in calculating the entrained air content, A, in accordance with Section 6.

Calculation:

6. The entrained air content of the concrete shall be calculated as follows:

$$A = A_1 - A_2 \qquad (1)$$

where:

A = entrained air content, percentage by volume of concrete,

A_1 = apparent air content, percentage by volume of concrete (Section 5), and

A_2 = aggregate correction factor, percentage by volume of concrete (Section 4).

APPENDIX I

THEORETICAL AND PRACTICAL CONSIDERATIONS UNDERLYING THE ROLLING METHODS OF MEASURING ENTRAINED AIR IN CONCRETE

(*a*) *Determination of Compression Factor, C:*

Before water is added over the concrete the air in the concrete sample is under the pressure exerted on it by the concrete, but after water is added the pressure is increased by the head of water measured from the surface of the concrete to the zero mark in the neck of the cover. This increase in pressure compresses the air and as a result the indicated air content determined by the rolling method is slightly lower than the actual air content. The compression factor, C, which is applied to correct for this effect, is determined as follows:

$$C = \frac{B + c + h}{B + c} \quad \ldots\ldots\ldots\ldots (1)$$

where:

B = barometric pressure (converted to inches of water by multiplying inches of mercury by 13.6),

c = average pressure (in inches of water) exerted by concrete on its entrained air (taken as head of concrete from surface of concrete to midheight of concrete in bowl),

The weight per cubic foot of the concrete will not vary enough to have a significant effect, and assuming it to be 150 lb., c will be equivalent to 1.2 times d in inches of water, where d = depth of concrete sample,

h = height of water column (in inches) from top surface of concrete to zero mark in cover.

An idea of the value of the compression factor, C, is given by the following illustration:

For a rolling apparatus with a bowl 8 in. deep, the value of d, c, and h will be about 8.0, 9.6, and 9.0 in. respectively. Assuming that the barometric pressure, B, will be about 407.2 in. of water at sea level, 339.0 in. at an elevation of 5000 ft., and about 250.0 in. at an elevation of 13,000 ft. above sea level, the value of C will be about 1.022 at sea level, 1.026 at an elevation of 5000 ft., and 1.035 at an elevation of 13,000 ft. above sea level. Variations of several inches from the values for d, c, and h will not have a significant effect on the value of C.

(*b*) *Determination of Alcohol Contraction Factor, R:*

Symbols and Equations.—It is convenient to use the following symbols and formulas:

V_a = volume of entrained air in concrete in cubic inches,

V_b = volume of isopropyl alcohol used to restore liquid level, in milliliters (may be converted to cubic inches by multiplying by 0.061).

V = volume of concrete sample, in cubic inches,

V_1 = volume of water added over concrete to zero mark in neck of cover, in cubic inches,

v = volume of water in concrete, in cubic inches

A = entrained air content, per cent by volume of concrete. Hence,

$$A = \frac{V_a}{V} \times 100 \quad \ldots\ldots\ldots\ldots (1)$$

A_1 = apparent air content (indicated by volume of alcohol used), per cent by volume of concrete. Hence,

$$A_1 = \frac{0.061 V_b}{V} \times 100 \quad \ldots\ldots\ldots (2)$$

p = volume of alcohol, V_b, in per cent of the sum of the volume of alcohol and volume of water, $(V_1 + v)$, to which it is added. Hence,

$$p = \frac{0.061 V_b}{0.061 V_b + (V_1 + v)} \times 100 \quad \ldots (3)$$

or, substituting for $0.061\ V_b$ its value obtained from Eq. (2), namely

$$\frac{A_1 V}{100},$$

$$p = \frac{100\ A_1 V}{A_1 V + 100\,(V_1 + v)} \quad \ldots\ldots (4)$$

r = contraction in volume of mixture in per cent of original volume of alcohol plus water as indicated by the curves in Fig 7(a) for given values of p.

In the following derivation of the alcohol-contraction factor, R, this factor represents the ratio of the volume of alcohol after mixing with water to the volume of alcohol added to the water. Although the contraction in volume is primarily a function of the mixture, it is convenient to consider that the water retains its original volume and that all the contraction which occurs is within the volume of alcohol. On this basis,

$$R = \frac{p - r}{p} = 1 - \frac{r}{p} \quad \text{........(5)}$$

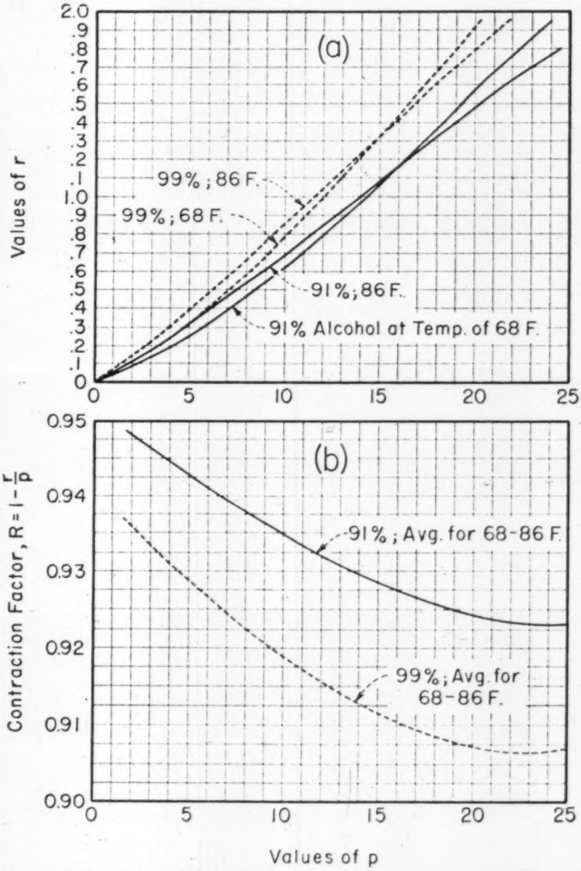

FIG. 7.—Values of r and R for Given Values of p. Data from Table II.

p = volume of isopropyl alcohol of a given strength in per cent of the sum of the volume of alcohol and volume of water to which it is added

r = contraction in volume of mixture in per cent of the original volume of alcohol plus water.

R = ratio of the volume of the alcohol after mixing with water to the volume of alcohol added to the water, assuming that all the contraction that results from mixing occurs within the volume of alcohol.

$$R = \frac{p - r}{p} = 1 - \frac{r}{p}$$

To obtain values of R, p must first be evaluated by means of Eq. 3 or 4. If by the design of the equipment the volume of water added over the concrete is about equal to the volume of the concrete sample, then $V_1 = V$. Also if the volume of water in the

TABLE I.—VALUES OF CONTRACTION FACTOR, R, FOR DIFFERENT VALUES OF APPARENT AIR CONTENT, A_1, AMOUNT OF WATER, $V_1 + v$, AND STRENGTHS OF ISOPROPYL ALCOHOL USED.

Values of p were calculated from Eq. 4 and values of R were taken from the curves shown in Fig. 7(b) for the given values of p

A_1	$V_1 + v = 1.1\ V$			$V_1 + v = 1.17\ V$			$V_1 + v = 1.3\ V$		
	p	R		p	R		p	R	
		91 per cent	99 per cent		91 per cent	99 per cent		91 per cent	99 per cent
2.0........	1.8	0.949	0.936	1.7	0.949	0.937	1.52	0.949	0.937
3.0........	2.7	0.947	0.934	2.5	0.947	0.935	2.3	0.948	0.935
4.5........	4.3	0.944	0.931	3.7	0.945	0.932	3.3	0.946	0.933
6.0........	5.2	0.942	0.928	4.9	0.943	0.929	4.4	0.944	0.930
9.0........	7.6	0.938	0.923	7.2	0.939	0.924	6.5	0.940	0.926
		(0.944)[a]	(0.930)		(0.945)	(0.931)		(0.945)	(0.932)
12.0........	9.8	0.935	0.919	9.3	0.936	0.920	8.5	0.937	0.921
15.0........	12.0	0.932	0.915	11.4	0.932	0.916	10.3	0.934	0.918
18.0........	14.4	0.929	0.912	13.3	0.930	0.913	12.2	0.932	0.915

[a] Values in parentheses are averages of the values of R for a range in A_1 of 2.0 to 9.0 corresponding to a range in entrained air content, A, of about 1.9 to 8.5 per cent.

TABLE II.—VALUES OF r AND R FOR GIVEN VALUES OF p

(Definitions of p, r, and R are given in notes for Fig. 7.

Alcohol per cent by volume, p	91 per cent Isopropyl Alcohol						99 per cent Isopropyl Alcohol						Average Value of R from 68 to 86 F. for	
	68 F. (20 C.)			86 F. (30 C.)			68 F. (20 C.)			86 F. (30 C.)				
	r	$\frac{r}{p}$	R	r	$\frac{r}{p}$	R	r	$\frac{r}{p}$	R	r	$\frac{r}{p}$	R	91 per cent Alcohol	99 per cent Alcohol
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)
2.0.......	0.09	0.045	0.955	0.12	0.060	0.940	0.11	0.055	0.945	0.15	0.075	0.925	0.948	0.935
3.5.......	0.17	0.048	0.952	0.22	0.063	0.937	0.21	0.060	0.940	0.27	0.077	0.923	0.945	0.932
5.0.......	0.25	0.050	0.950	0.32	0.064	0.936	0.32	0.064	0.936	0.39	0.078	0.922	0.943	0.929
10.0.......	0.62	0.062	0.938	0.69	0.069	0.931	0.78	0.078	0.922	0.85	0.085	0.915	0.935	0.912
15.0.......	1.07	0.071	0.929	1.08	0.072	0.928	1.32	0.088	0.912	1.33	0.088	0.912	0.929	0.912
20.0.......	1.58	0.079	0.921	1.48	0.074	0.926	1.93	0.096	0.904	1.81	0.090	0.910	0.924	0.907
25.0.......	2.04	0.081	0.919	1.84	0.073	0.927	2.44	0.097	0.903	2.22	0.089	0.911	0.923	0.907

concrete averages about 33 gal. per cu. yd. (range 26 to 40 gal. per cu. yd.), the volume of water is roughly equivalent to 17 per cent of the volume of the concrete. On this basis the volume of water, v, in the concrete will be equal to about 0.17 V and hence the total volume of water

$$V_1 + v = 1.17\ V \qquad (6)$$

and Eq. (4) becomes

$$p = \frac{100\ A_1}{A_1 + 117} \qquad (7)$$

Thus, after A_1 is determined, p can be found either from Eq. 4 or from the simpler Eq. 7, if the volume, V_1, of water added is equal to the volume, V of the concrete. Once p is calculated, r can be found from the curves in Fig. 7(a) and R can be computed from Eq. 5. As an alternative R can be found (for the same values of p) from curves such as those shown in Fig. 7(b). It should be pointed out that the curves in Fig. 7(b) give average values for R for the temperature range 68 to 86 F.

Factor R is 0.938 for Average Conditions of Test.—It is contemplated that apparatus for the suggested rolling and modified rolling methods of test will be so designed that the volume of water, V_1, added over the concrete will be about the same as the volume, V, of the concrete. In general, the water content, v, of the concrete will range from about 0.13 to 0.20 V and average about 0.17 V. It is also expected that isopropyl alcohol of either 91 or 99 per cent strength will be available and that most tests for entrained air content will be made on concrete having an actual air content between 2 and 8 per cent and a temperature between 68 and 86 F. Table I shows that the value of R is not appreciably influenced when the amount of water varies between 1.1 V and 1.3 V. The value of R decreases slightly with increase in apparent air content A_1 (or actual air content A) and also decreases as the strength of the alcohol is increased from 91 to 99 per cent. However, further study will show that no significant error in air content will be introduced if R is assumed to have a constant value of 0.938 for a range in:

A_1 from 2.0 to 9.0 per cent,
$V_1 + v$ from 1.1 V to 1.3 V,
Temperature from 68 to 86 F., and
Strength of isopropyl alcohol from 91 to 99 per cent.

For these reasons the value $R = 0.938$ can be used except for unusual departures from these average conditions.

Discussion of Table II.—Table II is based on specific gravity data for different strengths of isopropyl alcohol solutions given on p. 120, Vol. III, International Critical Tables.[1] The strengths of solutions given in these tables are based on the weight percentages of alcohol in the solution whereas commercial isopropyl alcohol is available in 91 and 99 per cent solutions by volume. Table II shows the calculated contractions which occur when different amounts of 91 and 99 per cent isopropyl alcohol are mixed with water. The first column represents the volume of alcohol (of a given strength) in percentage of the volume of alcohol plus the volume of water to which it is added. For example, the value of 2.0 in column 1 means that 2.0 ml. of alcohol (either of 91 or 99 per cent alcohol) are added to 98 ml. of water, so that the sum of the two separate volumes is equal to 100 ml. The value 0.09 for r in column 2 means that the separate volumes of 100 ml. will contract 0.09 ml. when the alcohol and water are thoroughly stirred and mixed. The value 0.045 in column 3 is the ratio of this contraction, 0.09 ml., in column 2 to the 2 ml. of alcohol shown in column 1. The value of 0.955 for R in column 4 is $1 - \frac{r}{p}$. It indicates that the volume of alcohol added minus the contraction in volume is equal to 0.955 times the volume of alcohol added in column 1.

Method of Calculating Contraction Values Given in Table II.—Example: 5 ml. of 91 per cent isopropyl alcohol (by volume) are added to 95 ml. of water at 68 F. Find contraction in volume of mixture.

From Critical Tables:
Density of 100 per cent alcohol at 68 F. = 0.7854 g. per milliliter
Density of water at 68 F. = 0.9982 g. per milliliter
Weight of 0.91 ml. of alcohol =
$0.91 \times 0.7854 = 0.714714$ g.
Weight of 0.09 ml. of water =
$0.09 \times 0.9982 = 0.089838$ g.

Combined weight of alcohol and water = 0.804552 g.

Weight of alcohol = $\frac{0.714714}{0.804552} \times 100 =$ 88.833 per cent of total weight.

From Critical Tables a solution of this strength has a density of 0.8125 g. per milliliter.
5 ml. of 91 per cent alcohol will then contain:
$5 \times 0.8125 \times 0.88833 =$ 3.60870 g. of alcohol
$5 \times 0.8125 \times 0.11167 =$ 0.45365 g. of water
When these 5 ml. of alcohol are added to 95 ml. of water (weighing $95 \times 0.9982 = 94.829$ g.) the solution will contain:

3.60870 g. of alcohol
0.45365 g. of water
94.82900 g. of water

98.89135 g. total weight

The weight of alcohol = $\frac{3.6087}{98.8913} \times 100 =$ 3.649 per cent of total weight

From Critical Tables a solution of this strength has a density of 0.9914 g. per ml.

Hence volume of 98.89135 g. will be

$$\frac{98.89135}{0.9914} = 99.75 \text{ ml.}$$

Then, contraction in volume, r, = 100.00 − 99.75 = 0.25 ml.

and $$\frac{r}{p} = \frac{0.25}{5.0} = 0.050$$

and $$R = \frac{p-r}{p} = 1 - \frac{r}{p} = 1 - 0.050 = 0.950$$

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[1] International Critical Tables of Numerical Data, Vol. III, First Ed., McGraw-Hill Book Co., New York, N.Y. (1928).

APPENDIX II

THEORETICAL AND PRACTICAL CONSIDERATIONS UNDERLYING THE PRESSURE METHOD OF MEASURING ENTRAINED AIR IN CONCRETE

(*a*) *Tests Indicate Conformity with Boyle's Law:*

The pressure method is based on the assumption that when pressure is applied to the concrete the composite volume of the minute cells of entrained air distributed throughout the concrete is reduced in conformity with Boyle's law and in the same proportion as that of a corresponding single volume of air to which pressure is applied directly. This implies that there is little or no restraint, such as particle interference within the mass of concrete or boundary friction between the concrete and the surface of the bowl, to reduce the effect of the applied pressure.

The validity of these assumptions had to be verified in some convincing way to inspire confidence in the accuracy and reliability of the results indicated by the pressure method. Such evidence was provided by extensive series of tests[1] which showed that the entrained air contents of fresh concrete samples tested by the pressure method, when corrected for the porosity of the aggregate, were practically the same as the air contents indicated by parallel tests with the rolling method (based on the volumetric principle for measuring air content) even when using widely different concretes and conditions of test. Comparative tests also showed good agreement for both the pressure and rolling methods with the less precise gravimetric method (A.S.T.M. Method C 138-44)[2] for determining the air content of fresh concrete. The general agreement between the results of procedures based on these three widely different principles of measuring air content provided substantial evidence that the change in volume of entrained air in concrete responds to pressure application in conformity with Boyle's law as assumed.[3]

(*b*) *Gage Pressure for Direct Indication of Air Content:*

From a practical standpoint it was highly desirable that the apparatus be designed to indicate percentage of air directly. To attain this objective either a very accurate pressure gage would have to be available and maintained to indicate when the proper theoretical pressure was being applied, or the desired reading on a less accurate pressure gage would have to be determined by calibrating the indicator against a known volume of air.

All things considered, it appears that the latter alternative is most practical and that the calibration procedure outlined in Section 3.(f) of the Suggested Pressure Method of Test, Part III (using a cylinder of known volume) is probably the most direct and reliable basis for determining and checking the proper operating pressure under the conditions existing at any given job location. This procedure eliminates the need for accurate pressure application in terms of pounds per square inch or other units so long as the needle of the pressure gage is brought to the same point as that determined in the calibration test. Any suspected change in the functioning of the gage needle (as might occur during shipping and handling of the equipment, etc.) can be checked promptly by recalibrating to determine the correct operating pressure.

Figs. 8(*a*) and 8(*b*) illustrate the parallelism between the test for entrained air in a concrete sample and the test for a known volume of air introduced by the calibration cylinder. The parallelism is not perfect either initially or at later stages of the test, largely because the entrained air in a sample of fresh concrete is distributed and initially subjected to atmospheric pressure, plus an average pressure head, c, of concrete (equivalent to about 9.6-in. head of water), whereas the air introduced by the calibration container is concentrated and initially subjected to atmospheric pressure plus a pressure head of $r - t_1$ (about 8.0-in. head of water) when the bowl is level full of water. However, it can be shown from the detailed analysis and parallel derivations presented under cases 1 and 2 below that for the characteristics of this apparatus the theoretical pressure, P, which must be applied to a sample of concrete for direct indication of a given percentage of air in the concrete will at sea level be only about 0.13 psi. lower than the corresponding pressure which would be required for the same percentage of air introduced by the calibration cylinder. Moreover, this difference decreases progressively with decrease in barometric pressure and is only about 0.05 psi. at 13,000 ft. above sea level. From this analysis it was concluded that, in general, the correct operating pressure for testing concrete will be the pressure determined by the calibration test, minus 0.10 psi. as given in Eq. 5, Section 3(j) of the Suggested Pressure Method of Test (Part III.)

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[1] Comparison of results by different methods are illustrated in Fig. 6, 8, and 9 of paper on "Development and Study of Apparatus and Methods for the Determination of Air Content of Fresh Concrete," see footnote 4 on p. 834.

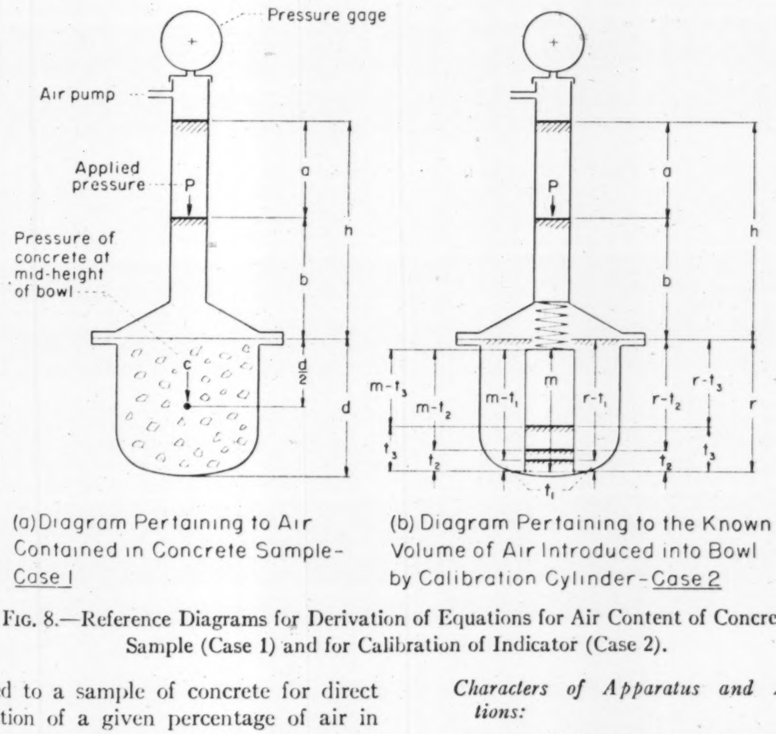
[2] In the gravimetric method the sum of the absolute volumes of the ingredients in a known volume of concrete is calculated and subtracted from the known volume, the difference being taken as the volume of air in the concrete.

[3] Except possibly with stiff concretes of very low slump and low air content.

(*c*) *Derivations:*

Case 1 (*Fig. 8*(*a*)).

Object: To determine the theoretical pressure, P, which must be applied to a sample of concrete so that each unit (inch) on the graduated precision bore tube of indicator will represent one per cent air in the concrete.



(a) Diagram Pertaining to Air Contained in Concrete Sample-Case 1

(b) Diagram Pertaining to the Known Volume of Air Introduced into Bowl by Calibration Cylinder-Case 2

FIG. 8.—Reference Diagrams for Derivation of Equations for Air Content of Concrete Sample (Case 1) and for Calibration of Indicator (Case 2).

Characters of Apparatus and Assumptions:

V = level full capacity of flanged bowl in cubic inches,

e = cross-sectional area of water column in square inches,

a = change in height of water column (in inches) when pressure, P, is applied,

b = height of water column (in inches) above top of concerte (flange of bowl) when pressure, P, is applied,

h = height of water column (in inches) from top surface of concrete (flange or bowl) to zero mark. $h = a + b$,

d = height of bowl in inches, and
c = average pressure (in inches of water) exerted by concrete on its entrained air (taken as head of concrete from mid-height of bowl). The weight per cu. ft. of concrete will not vary enough to hava a significant effect, and assuming it at 150 lb., c will be equivalent to a head of 1.2 times d inches of water.

Symbols Used in Derivation:

A = air content of concrete in bowl, per cent by volume of bowl. Hence, $A = 100 \times \frac{V_1}{V}$
B = barometric pressure (converted to inches of water by multiplying inches of mercury by 13.6),
P = applied pressure, in inches of water,
P_1 = average absolute pressure on air in concrete sample contained in bowl. Hence, $P_1 = B + c$,
P_2 = average absolute pressure on air in concrete sample when water column is at zero mark. Hence, $P_2 = P_1 + h = B + c + h$,
P_3 = average absolute pressure on air in concrete sample under partial head of water, b, and applied pressure, P, (water level is lowered when air in concrete is compressed). Hence, $P_3 = B + c + b + P$,
V_1, V_2, and V_3 = volume of air in concrete sample under pressure P_1, P_2, and P_3, respectively.

In the following derivation it has been assumed (1) that application of pressure, P, causes no expansion in volume of the assembled indicator, and (2) that no restraint is set up within the concrete or at its contact with the surface of the bowl which would limit the effect of the applied pressure, P.

From Boyle's Law:

$$\frac{P_1}{P_2} = \frac{V_2}{V_1} \quad \text{or} \quad V_2 = \frac{P_1 V_1}{P_2} \quad \ldots\ldots (1)$$

and,

$$\frac{P_1}{P_3} = \frac{V_3}{V_1} \quad \text{or} \quad V_3 = \frac{P_1 V_1}{P_3} \quad \ldots\ldots (2)$$

As the reduction in volume of air in the concrete resulting from the application of pressure, P, is equal to the volume of the water column between the zero mark and the lowered water level, then

$$ea = V_2 - V_3 \quad \ldots\ldots\ldots\ldots (3)$$

Substituting Eqs. 1 and 2 in Eq. 3,

$$ea = \frac{P_1 V_1}{P_2} - \frac{P_1 V_1}{P_3}$$

Rearranging

$$P_3 = \frac{P_1 P_2 V_1}{P_1 V_1 - P_2\, ea} \quad \ldots\ldots\ldots (4)$$

From diagram (*a*) of Fig. 8,

$$P_3 = P_1 + b + P \quad \ldots\ldots\ldots\ldots (5)$$

Substituting Eq. 5 in Eq. 4,

$$P = \frac{P_1 P_2 V_1}{P_1 V_1 - P_2\, ea} - b - P_1 \quad \ldots\ldots (6)$$

Since $V_1 = \frac{AV}{100}$, then Eq. 6 becomes

$$P = \frac{P_1 P_2 A V}{P_1 A V - 100\, P_2\, ea} - b - P_1 \quad \ldots (7)$$

By construction of equipment the graduations on the water column are such that each linear inch of the water column is to represent one per cent of air in the concrete.

$$\text{Then } a = A \quad \ldots\ldots\ldots\ldots (8)$$

Substituting Eq. 8 in Eq. 7,

$$P = \frac{P_1 P_2 V}{P_1 V - 100\, P_2\, e} - b - P_1 \quad \ldots (9)$$

From diagram (*a*) of Fig. 8,

$$P_1 = B + c \quad \ldots\ldots\ldots\ldots (10)$$

$$P_2 = P_1 + h = B + c + h \quad \ldots\ldots (11)$$

$$b = h - a = h - A, \text{ from Eq. 8} \quad \ldots (12)$$

Substituting Eqs. 10, 11 and 12 in Eq. 9

$$P = \frac{(B + c)(B + c + h)V}{(B + c)V - 100\, e\, (B + c + h)} - B - c - h + A \quad \ldots (13)$$

In Eq. 13 the pressures, P and B, are in inches of water, and P can be converted to pounds per square inch by multiplying by 0.0361. Hence,

$$P \text{ (in psi.)} = 0.0361 \left[\frac{(B+c)(B+c+h)V}{(B+c)V - 100\,e\,(B+c+h)} - B - c - h + A \right] \ldots (14)$$

For the 0.22-cu. ft. indicator shown in Fig. 5, $h = 12.0$ in.; $d = 8.0$ in.; $c = 9.6$ in. (of water) and $e = 1.217$ sq. in.

and when these values are substituted in Eq. 14, then

$$P \text{ (in psi.)} = 0.0361 \left[\frac{(B+9.6)(B+21.6)V}{(B+9.6)V - 121.7\,(B+21.6)} - B - 21.6 + A \right] \ldots (15)$$

when $B = 407.2$ in. of water (29.92 in. of mercury), and

$V = 388$ cu. in.

$$P \text{ (in psi.)} = 7.33 + 0.0361\,A \ldots (16)$$

and when $B = 250.0$ in. of water (18.40 in. of mercury), and

$V = 388$ cu. in.

$$P \text{ (in psi.)} = 4.76 + 0.0361\,A \ldots (17)$$

Case 2 (Fig. 8(b)):

Object: To determine the theoretical pressure P which must be applied to the known volume of air in the calibration cylinder so that each unit (inch) on the graduated precision bore tube of indicator will represent one per cent air introduced by the calibration cylinder.

Characteristics of Apparatus and Assumptions:

V = level full capacity of flanged bowl in cubic inches,

e = cross-sectional area of water column in square inches,

a = change in height of water column (in inches) when pressure P is applied,

b = height of water column (in inches) above top of flange of bowl when pressure P is applied,

h = height of water column (in inches) from top of flange of bowl to zero mark. $h = a + b$,

r = distance between top of flange of bowl and rim of inverted calibration cylinder in inches,

k = internal cross-sectional area of calibration cylinder in square inches,

m = inside depth of calibration cylinder,

t_1 = rise of water in calibration cylinder under a head of water equal to $r - t_1$ when bowl is level full of water, in inches,

t_2 = rise of water in calibration cylinder under a head of water equal to $r - t_2 + h$, when water column is at zero mark, in inches,

t_3 = rise of water in calibration cylinder under a head of water equal to $r - t_3 + b + P$, in inches,

Symbols Used in Derivation:

A = air content of calibration cylinder when flanged bowl is level full of water, per cent by volume of bowl. Hence,

$$A = 100 \times \frac{V_1}{V}$$

B = barometric pressure (converted to inches of water by multiplying inches of mercury by 13.6),

P = applied pressure, in inches of water,

P_1 = absolute pressure on air in calibration cylinder when bowl is level full of water. Hence,

$$P_1 = B + (r - t_1)$$

P_2 = absolute pressure on air in calibration cylinder when water column is at zero mark. Hence, $P_2 = B + (r - t_2) + h$,

P_3 = absolute pressure on air in calibration cylinder when pressure P is applied. Hence, $P_3 = B + (r - t_3) + b + P$,

V_1, V_2, and V_3 = volume of air in calibration cylinder under pressure P_1, P_2, and P_3 respectively.

Equation (7) for P which follows was derived by the same procedure as used in Eqs. 1 to 6 inclusive in case 1. Here, as in case 1, it was assumed that the application of pressure causes no expansion in volume of the assembled indicator.

$$P = \frac{P_1 P_2 A V}{P_1 A V - 100 P_2 e a} - B - r + t_3 - b \quad (7)$$

By construction of equipment the graduations on the water column are such that each linear inch of the water column is to represent 1 per cent of air introduced by the calibration cylinder. Then,

$$a = A \ldots\ldots\ldots\ldots\ldots (8)$$

Substituting Eq. 8 in Eq. 7,

$$P = \frac{P_1 P_2 V}{P_1 V - 100 P_2 e} - B - r + t_3 - b \quad (9)$$

From diagram (*b*) of Fig. 8,

$$P_1 = B + r - t_1 \ldots\ldots\ldots (10)$$
$$P_2 = B + r - t_2 + h \ldots\ldots (11)$$
$$b = h - a = h - A, \text{ from Eq.8} \ldots (12)$$

Substituting Eqs. 10, 11, and 12 in Eq. 9,

$$P = \frac{(B + r - t_1)(B + r - t_2 + h)V}{(B + r - t_1)V - 100\,e\,(B + r - t_2 + h)} - B - r - h + t_3 + A \,.. (13)$$

In Eq. 13 the pressures P and B are in inches, and P can be converted to pounds per square inch by multiplying by 0.0361. Hence,

$$P \text{ (in psi.)} = 0.0361 \left[\frac{(B + r - t_1)(B + r - t_2 + h)V}{(B + r - t_1)V - 100\,e\,(B + r - t_2 + h)} - B - r - h + t_3 + A \right] .. (14)$$

For the 0.22 cu. ft. indicator shown in Fig. 5,

$$h = 12.0 \text{ in.}, r = 8.15 \text{ in.}, m = 7.44 \text{ in.}, \text{ and } A = 6.62 \text{ in.}$$

It can be shown that the values for t_1, t_2, and t_3 are related to these constants[4] and vary only slightly with a wide range in barometric pressure from 407.2 in. of water to 250 in. of water (29.92 to 18.40 in. of mercury). For these conditions, representing a range in altitude from sea level to about 13,000 ft. above sea level, t_1 will range from 0.143 to 0.228 in., t_2 from 0.344 to 0.541 in., and t_3 from 2.63 to 2.81 in. No appreciable error will be introduced by using values of $t_1 = 0.15$ in.; $t_2 = 0.35$ in., and $t_3 = 2.65$ in., in Eq. 14. Then,

$$P \text{ (in psi.)} = 0.0361 \left[\frac{(B + 7.9)(B + 19.7)V}{(B + 7.9)\,V - 121.7\,(B + 19.7)} - B - 17.4 + A \right] .. (15)$$

When $B = 407.2$ in. of water (29.92 in. of mercury), and
$V = 388$ cu. in.,
P (in psi.) $= 7.46 + 0.0361\,A \ldots (16)$
And when $B = 250.0$ in. of water (18.40 in. of mercury), and
$V = 388$ cu. in.,
P (in psi.) $= 4.81 + 0.0361\,A \ldots (17)$

APPENDIX III

CONSIDERATIONS INVOLVED IN THE AGGREGATE CORRECTION FACTOR FOR PRESSURE METHOD OF MEASURING ENTRAINED AIR IN CONCRETE

(*a*) *Comments Regarding Test for Aggregate Correction Factor on Combined Fine and Coarse Aggregate:*

The procedure for determining the aggregate correction factor given in Section 4 of the Suggested Pressure Method of Test (Part III) is given in general terms so that it will apply to any given combination of fine and coarse aggregate that may be present in the concrete sample to be tested for entrained air content. For this reason the tests are based on the weights of fine and coarse aggregate computed by equations (See Eqs. 1 and 2) and scales would be required to weigh the samples.

There are, however, many concreting operations such as on highway construction jobs where the proper amount of fine and coarse aggregate needed for the aggregate-correction factor can be obtained without scales by using the corresponding inundated volumes. Moreover, it can be shown that for most pavement concretes employing fine and coarse aggregate no significant error will be introduced by making all aggregate correction-factor tests on the amounts of fine and coarse aggregates as they occur in concrete samples of average composition instead of using the exact volumes of fine and coarse aggregate as they occur in concrete samples of substantially leaner or richer composition. These points can be illustrated by the following example:

Calculations indicate that a 0.22 cu. ft.

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[4] These relaions are given by the following expressions:

$$t_1^2 - (B + r + m)\,t_1 + mr = 0 \ldots\ldots\ldots (18)$$
$$t_2^2 - (B + r + m + h)\,t_2 + m\,(r + h) = 0. \;(19)$$
$$t_3^2 - (P + B + r + m + h - a)\,t_3 + m\,(P + r + h - a) = 0 \;\ldots\ldots\ldots\ldots (20)$$

The value t_1 is useful in determining the factor Π which enters into the calibration factor K discussed in Section 3.(e) of the Suggested Pressure Method of Test (Part III).

In general,

$$\Pi = \frac{m - t_1}{m} \ldots\ldots\ldots\ldots\ldots\ldots (21)$$

where Π represents the ratio of air in calibration cylinder after inundation to volume before inundation under a head of $r - t_1$ inches of water.

sample of average paving type concrete containing 4 per cent air, 35 per cent sand (by weight of total aggregate) and 6.0 sack cement per cubic yard will contain about 137 cu. in. of dry sand (about 130 cu. in. or about 95 per cent of dry volume after inundation) and about 277 cu. in. of coarse aggregate. In leaner concrete (4.5 sack, 37 per cent sand), the total volume of the fine and coarse aggregate, either on a dry or inundated basis, will be about 3 per cent higher than for the usual six-sack concrete and in richer concrete (7.5 sack, 33 per cent sand) the volume of the aggregate will be about 5 per cent lower than for the usual six-sack concrete. However, these differences between the volume of aggregate in rich, lean, and medium concretes are so small in their effect on the aggregate correction factor that they usually can be ignored and no significant errors are introduced if all aggregate correction factors are based on inundated volumes of fine and coarse aggregates corresponding to the amounts present in the average mix, as assumed above. As a matter of fact, it will require an error of 10 per cent in the total amount of aggregate in the test sample to cause an error corresponding to 0.05 per cent air if the true aggregate correction factor is 0.50 per cent air.

In view of the preceding paragraphs, the procedure outlined in Section 4.(b) and 4.(c) of the Suggested Pressure Method of Test (Part III) could be based on inundated volumes of fine and coarse aggregate corresponding to the weights w_f and w_c normally present in the sample of fresh concrete whose entrained air content is to be determined.

(*b*) *Determination of Aggregate Correction Factor from Tests on Separate Samples of Fine and Coarse Aggregate:*

Sometimes it may be more convenient or desirable to determine the aggregate correction factor from tests on separate samples of fine and coarse aggregate either in the same amounts and moisture contents as they occur in the concrete test sample, or in other amount and moisture contents. Such information will indicate the characteristics of a given fine or coarse aggregate well in advance of the concreting operations on highway construction or other jobs and will show when, if, and to what extent special care must be exercised to obtain reliable determinations of the entrained air in concrete.

In using the data obtained in separate tests on the fine and coarse aggregate, care must be taken to include the effect of the expansion factor for the indicator used.[1] This effect is believed to be properly considered in the calculation of the characteristics F and C, the partial aggregate factors X and Y, and of the aggregate correction factor Z for combined fine and coarse aggregate.

Procedure (suitable for indicator of 0.20 or 0.22 cu. ft. capacity):

(*a*) In a separate container inundate 20 lb. of a representative sample of fine aggregate in water,

(*b*) Allow the aggregate to absorb water for 5 min. to bring it to a moisture content comparable to that which obtains at the time the concrete is normally sampled, and drain off the excess water,

(*c*) Fill the indicator bowl a third full of water and gradually add the fine aggregate, a small scoopful at a time, until the entire sample has been inundated in the bowl. Add each scoopful in a manner which will entrap as little air as possible and remove all accumulations of foam promptly. Also tap sides of bowl and rod the upper inch of the aggregate after the addition of each scoopful of aggregate to eliminate entrapped air,

(*d*) When all aggregate has been placed in the bowl, strike off all foam and excess water and clean flanges of both bowl and conical cover. Then clamp on cover, fill with water, apply the correct operating pressure, P, and read and record the maximum (h_1) and minimum (h_2) readings for two cycles of pressure application and release as described in Sections 5.(b) and 5.(c) of the Suggested Pressure Method of Test (Part III).

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[1] The expansion factor is discussed in Section 3 (d) of the Suggested Pressure Method of Test (Part III).

(*e*) Follow the same procedure for a 20-lb. sample of coarse aggregate.

(*f*) The characteristics of the fine and coarse aggregate sample represented by the values F and C are calculated as indicated in Table III.

(*g*) The characteristics F and C are used to calculate the aggregate correction factor Z for any combination of fine and coarse aggregate that may be present in a concrete sample to be tested for entrained air content.

TABLE III.—CALCULATION OF CHARACTERISTICS F AND C (20-LB. SAMPLES).

Cycle	Applied gage pressure, (a) psi.	Readings of water level		
			Fine aggregate	Coarse aggregate
1.......	P............. Zero..........	Maximum (h_1) Minimum (h_2)	—— ——	—— ——
		Difference ($h_1 - h_2$)	——	——
2......	P............. Zero..........	Maximum (h_1) Minimum (h_2)	—— ——	—— ——
		Difference ($h_1 - h_2$)	——	——
Average Difference (Cycles 1 and 2)....................			F_1 = ——	C_1 = ——
Expansion Factor for Indicator[b]			D_f = ——	D_c = ——
Net Difference, Characteristic			$F = F_1 - D_f$ = ——	$C = C_1 - D_c$ = ——

[a] The applied gage pressure P is the same as that determined by the calibration test in Section 3.(f) of the suggested Pressure Method of Test (Part III).

[b] D_f and D_c are the expansion factors for the indicator or indicators that may be used in the tests.

$$Z = X + Y + D \ldots\ldots\ldots (1)$$

where

X = the partial aggregate correction factor for the fine aggregate of weight w_f in the concrete sample,[2]

Y = the partial aggregate correction factor for the coarse aggregate of weight w_c in the concrete sample,[2]

D = expansion factor for the indicator used in testing the concrete sample for air content.[3]

In general

$$X = F \times \frac{w_f}{w_{f_1}} \ldots\ldots\ldots\ldots (2)$$

$$Y = C \times \frac{w_c}{w_{c_1}} \ldots\ldots\ldots\ldots (3)$$

where

w_f = pounds of fine aggregate in concrete sample under test,

W_{f_1} = pounds of fine aggregate used in test for characteristic F,

F = value of characteristic of fine aggregate of weight W_{f_1},

w_c = pounds of coarse aggregate in concrete sample under test,

W_{c_1} = pounds of coarse aggregate used in test for characteristic C,

C = value of characteristic of coarse aggregate of weight W_{c_1}.

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[2] The weights w_f and w_c may be determined by Eqs. 1 and 2 given in Section 4 (b) of the Suggested Pressure Method of Test (Part III).

[3] The expansion factor for the indicators developed by the Research Laboratories of the Portland Cement Assn. corresponds to about 0.05 per cent air for 0.22-cu. ft. capacity, about 0.02 per cent air for 0.44-cu. ft. capacity, and 0.05 per cent air for 2.8-cu. ft. capacity, but the term D is included in Eq. 1 for Z because indicators are being used where the expansion factors corresponds to 0.20 to 0.30 per cent, air and must not be ignored.

INDIANA METHOD FOR MEASURING ENTRAINED AIR IN FRESH CONCRETE*

By P. D. Miesenhelder[1]

Synopsis

This paper is devoted principally to a description of the so-called "Indiana Method," for determining the quantity of entrained air in concrete, making use of the relationship between the unit weights of a concrete sample with and without air. The method has advantages and weaknesses that are described, together with some comparison with other methods for measuring entrained air.

With the advent of air-entraining cement, which was first used to any extent in Indiana during the construction season of 1945, it became necessary to devise a test method suitable for field use to determine the quantity of air in the fresh concrete. In 1945 the use of air-entraining cement was permitted with the hope that the concrete would contain not less than 3 nor more than 5 per cent air. In 1946 air-entraining cement was required to be used and the air content of the concrete was specified as 3 to 5 per cent.

Somewhat prior to 1945 some experimental work was conducted in the laboratory to determine the quantity of air in concrete using procedure and equipment similar to that specified in the A.S.T.M. Tentative Method of Test for Air Content (Volumetric) of Freshly Mixed Concrete (C 173 - 42 T).[2] We had a distinct preference for this method, in which the unit weight of the air-free concrete is determined experimentally rather than by computation involving the sum of the absolute volumes of all constituent materials, as described in A.S.T.M. Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138 - 44).[3] In devising the so-called "Indiana Method" for use in the field, it was desirable to make the procedure simple, quick, and easy to understand by the average inspector; and, because materials and equipment were virtually impossible to obtain at that time, it was necessary to make use of existing equipment with which the inspectors were already supplied.

The method is based on the equation

$$\text{Percentage of air} = \frac{(T - A)100}{T}$$

where:

T = unit weight of the air-free concrete, and

A = unit weight of the concrete containing air.

This equation is, of course, applicable regardless of the methods employed for determining T and A. The relation between percentage of air, T, and A is mathematically sound and, if T and A are determined accurately, the computed value for percentage of air will be accurate.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

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[2] 1946 Book of A.S.T.M. Standards, Part II, p. 1411.

[3] *Ibid.*, p. 513.

PROCEDURE IN CONDUCTING THE TEST

The unit weight of the concrete containing air A, is determined by the same procedure as that employed in the Method C 138 – 44, using a 0.5-cu. ft. cast aluminum yield bucket and platform scales graduated in ounces and sensitive to $\frac{1}{2}$ oz. The unit weight of the air-free concrete, T, is determined experimentally by measuring the volume, by displacement in water, of a sample of fresh concrete of known weight from which the air has been removed by washing. The principle involved is the same as that employed in Method C 173 – 42 T and is so old that no originality is claimed. In this determination the equipment used consists of the same scales and yield bucket used in the determination of A, plus a hook gage which converts the yield bucket into a pycnometer, the volume of which has been calibrated with water of known temperature. An accurate determination of T requires, of course, that all the air be released from the concrete sample and from the water in which the sample is immersed.

After the determination of A, the operator removes concrete from the yield bucket until approximately 30 lb. remain. The gross weight of the yield bucket and concrete sample is obtained and recorded on the field data sheet. Sufficient water is then added to immerse the sample to a depth of a few inches and the concrete washed by stirring with a $\frac{5}{8}$-in. diameter rod to remove the air. The hook gage is set in position, on reference points on the upper rim of the yield bucket, and more water added until the hook-gage impression on the water surface forms and breaks. The hook gage is then removed and the gross weight of the yield bucket and its contents is determined. From this, the weight recorded on the field data sheet is subtracted to obtain the weight of water which, with the concrete sample, fills the pycnometer to the hook-gage point. This weight of water is converted into cubic feet and subtracted from the volume of the pycnometer to obtain the volume of the air-free concrete sample in cubic feet. T is obtained by dividing the weight of the sample by its volume. Substitution in the formula gives the percentage of air. The necessary data and computations for a complete air determination test consist of four weighings and simple arithmetic.

ADVANTAGES OF THE METHOD

The "Indiana Method" has the advantage of simplicity of procedure and simplicity of equipment. The equipment is relatively inexpensive, not easily damaged, and easy to clean. When an entrained-air test is conducted, all the necessary data for a yield test are obtained, so the equipment and test serve a dual purpose. This is quite an advantage because the inspector is not burdened with additional equipment which serves only to measure entrained air. The procedure and computations are so simple that the average inspector can learn to conduct the test with very little instruction.

CRITICISMS OF THE METHOD

There are two weaknesses in the method, the necessity for accurate weighing and the lack of assurance that all the air has been removed. The scales are sensitive to $\frac{1}{2}$ oz., but unless the scale weights and beam are kept clean and the weighings are unaffected by wind this accuracy is fictitious. The most significant weighing is the gross weight of the yield bucket, concrete sample, and water to the hook-gage point. A mistake of 1 oz. in this weight produces an error in the computed percentage of air of 0.5. Insufficient washing of the sample may occur because of the tendency among inspectors to quit stirring too soon, in which case the presence of air still remaining in the sample will make the value of T too low. It has been our practice to skim the foam from the water surface after an initial stirring, stir again and skim off foam, and continue this procedure until foam no longer appears, which is assumed to be an indication that all the air has been removed. If the concrete contains slag or crushed stone as coarse aggregate, the stirring is somewhat more difficult than it is with gravel due to the interlocking of the angular-shaped pieces. For that reason it is likely that the test method requires more attention with such coarse aggregates. This discussion of thorough washing concerns the determination of T. Accuracy is also required in the determination of A since percentage of air is a function of both T and A. Accurate determinations of both T and A require, of course, that the sample be truly representative of the concrete. Even if the sample is perfect in this respect, thorough washing is necessary in determining T while proper compaction of the sample in the yield bucket is necessary in determining A. Although the placing of concrete in an 0.5-cu. ft. container is standard procedure, A is subject to a certain amount of variation. However, this criticism with reference to the constancy of A is unavoidable, and it is quite likely that A varies more widely in different parts of a structure or pavement than it does in a series of determinations.

COMPARISON WITH OTHER METHODS

In a series of tests under way at present, several methods for measuring entrained air are being studied. These include the "Indiana Method" with one set of equipment, the pressure method with two air meters, and a rolling method with three sizes of rolling containers, thereby affording six independent determinations of entrained air on each batch of concrete. In the rolling method, the concrete sample is washed by rolling instead of stirring. Each rolling container consists of a cylindrical base fitted with a conical lid at the top of which is a 3-in. pipe nipple with a zero mark near the top and a drain valve near the bottom. The volume between the zero mark and the drain, called V, is determined with water of known temperature. In this method the sample of concrete is placed in the bottom of the cylindrical base, rodded in the usual manner, and struck off to a given volume with a circular motion of a sheet metal template. Water is then introduced to the zero mark in the pipe nipple and immediately drawn off through the drain valve into a small can and saved. A cap is placed on the pipe nipple and the container placed on its side and rolled, at the conclusion of which the cap is removed and the water in the can replaced in the container. The drain valve is then opened and the water caught in a graduated cylinder. If this volume of water is called R, it will be seen that the percentage of air is equal to $(V-R)$ divided by the volume of the concrete sample. Results available to date indicate a fair agreement among the various methods, but the entire working range of air content has not yet been covered.

In order to afford a more detailed

comparison of the six tests than that afforded by a comparison of the six final results—percentages of air—and as a possible means of accounting for any variations that might occur in final results, the unit weights A of the six samples of concrete, all from the same batch, are being determined by weighing each apparatus before and after introducing the sample. If T is constant, A and percentage of air should vary inversely in a straight-line relation regardless of the test method, even though a knowledge of A is not needed in the pressure and rolling methods. However, we have been surprised to find that the six determinations of A on a single batch of concrete were not consistent with the six determinations of percentage of air. This seeming inconsistency is best described by an example. One batch of concrete was shown to contain 4.5 per cent air by one testing device and 4.7 per cent by another, with the results of the other four devices within this range, while the six determinations of A varied from 144.8 to 147.4 lb. per cu. ft. Possibly T varied in the six devices due to poor sampling of the batch, but it seems unlikely that the variation would occur in such a manner as to show almost exactly the same percentage of air by all six devices.

CONCLUSIONS

The test method described in this paper has now been in use by the State Highway Commission of Indiana with apparently satisfactory results during two construction seasons on fifteen contracts totalling approximately 78 miles of standard width pavement. The method has the advantages and disadvantages mentioned, but the principle employed is mathematically sound, and if the test is conducted properly the results will afford a practical measure of the quantity of air entrained in the concrete.

THE MEASUREMENT OF AIR ENTRAINED IN CONCRETE*

BY JOHN H. SWANBERG[1] AND T. W. THOMAS[1]

SYNOPSIS

This work was undertaken for the purpose of studying the pressure method for the determination of the air content of plastic concrete. The percentage of air as determined by the pressure method was compared to that obtained by the unit weight method. The data from 75 tests show an average air content 0.10 per cent higher by the pressure method. The work involved the study of three gage pressures and five different aggregates. Good agreement in results among the three gage pressures was obtained.

The adoption of air-entrained concrete as standard practice in paving by the Minnesota Department of Highways made it necessary that a means of determining the air content be found which would be accurate and convenient to use, particularly in the field. On the first projects involving the use of this type of concrete (built in 1940 and 1941) this determination was made in accordance with the A.S.T.M. Standard Method of Test for Yield of Concrete (C 138–39).[2] It was difficult to obtain consistent results with this method and it did not lend itself readily to field observations.

A description was obtained of the pressure method, first suggested by W. H. Klein and upon which further work was done by Stanton Walker. Apparatus of this type was built in the Department shop, and laboratory tests were made to determine its suitability. On the basis of these tests, modifications were made in the construction of the apparatus. The "air meter," as modified (shown in Fig. 1), was used on all paving projects constructed in 1946. The equipment as shown weighs approximately 45 lb. and

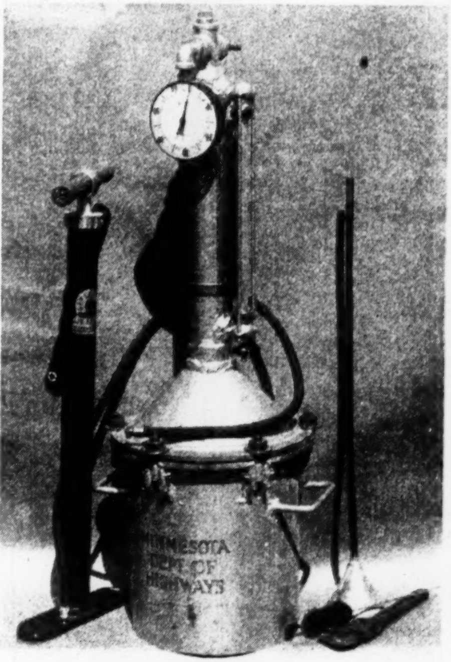

FIG. 1.—Air Meter.

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*Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Engineer of Materials and Research, and Research Engineer, respectively, Minnesota Department of Highways, St. Paul, Minn.

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 344.

yields consistent and apparently accurate results. The test can be made in approximately 5 min., and the equipment is such that it can be conveniently carried on the concrete finishing machine and be readily available to the inspector at all times.

the water level. The latter has the following advantages:

1. It permits rugged construction of the vertical column.

2. It permits easy reading since the water column is entirely in the clear.

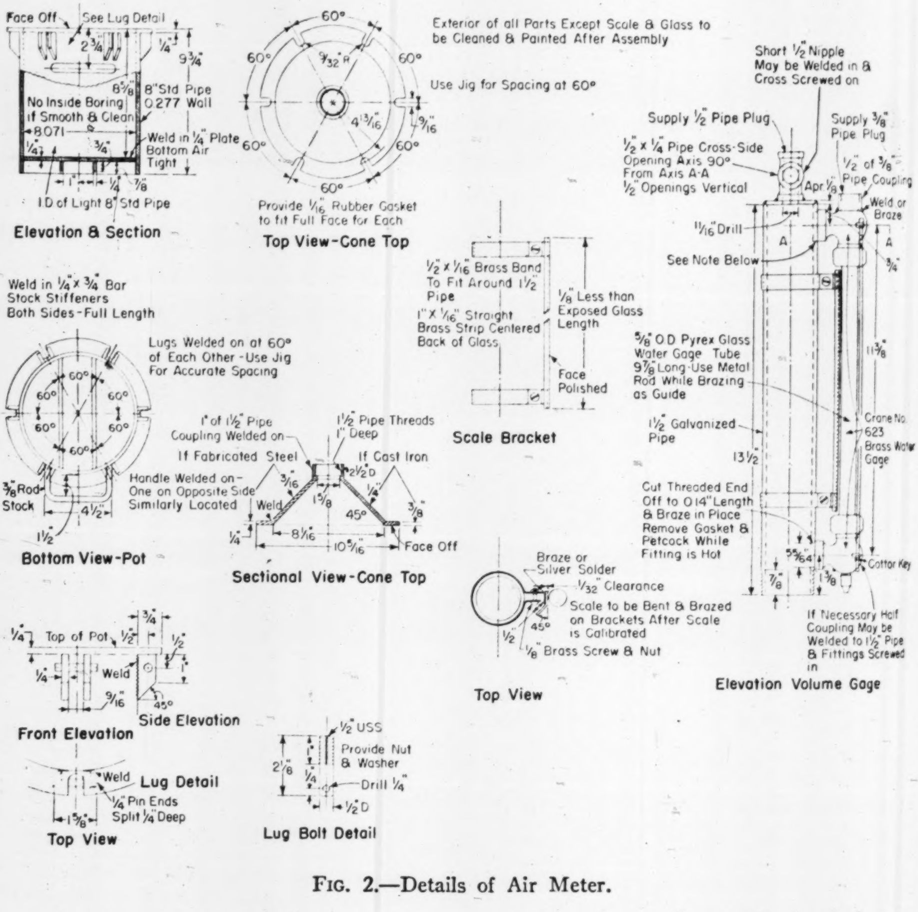
Face Off
See Lug Detail
2¾
¼"
9¾"
8⅝"
8"Std Pipe 0.277 Wall
No Inside Boring if Smooth & Clean
8.071
Weld in ¼" Plate Bottom Air Tight
¼"
¾"
1"
¼
⅞"
I.D of Light 8" Std Pipe
Elevation & Section
60°
9/32" R
4 13/16"
9/16"
Provide 1/16" Rubber Gasket to fit Full Face for Each
Top View-Cone Top
Exterior of all Parts Except Scale & Glass to be Cleaned & Painted After Assembly
Use Jig for Spacing at 60°
Short ½" Nipple May be Welded in & Cross Screwed on
Supply ½" Pipe Plug
Supply ⅜" Pipe Plug
½" x ¼ Pipe Cross-Side Opening Axis 90° From Axis A-A ½" Openings Vertical
Apr ⅛"
½" of ⅜" Pipe Coupling
Weld or Braze
11/16" Drill
A
See Note Below
¾"
½" x 1/16" Brass Band To Fit Around 1½" Pipe
1" X 1/16" Straight Brass Strip Centered Back of Glass
⅛" Less than Exposed Glass Length
Face Polished
Scale Bracket
⅝" O.D Pyrex Glass Water Gage Tube 9⅞" Long-Use Metal Rod While Brazing as Guide
11⅜"
1½" Galvanized Pipe
Crane No. 623 Brass Water Gage
13½"
Cut Threaded End Off to 0.14" Length & Braze in Place Remove Gasket & Petcock While Fitting is Hot
55/64"
⅞"
1⅜"
Cottor Key
If Necessary Half Coupling May be Welded to 1½" Pipe & Fittings Screwed in
Elevation Volume Gage
Weld in ¼" x ¾" Bar Stock Stiffeners Both Sides-Full Length
Lugs Welded on at 60° of Each Other-Use Jig For Accurate Spacing
⅜"Rod Stock
4½"
1½"
Handle Welded on-One on Opposite Side Similarly Located
Bottom View-Pot
1" of 1½" Pipe Coupling Welded on
1½" Pipe Threads 1" Deep
If Fabricated Steel
If Cast Iron
2½"D
3/16
¼"
⅜"
1⅜
Weld
45°
8 1/16
10 9/16"
Face Off
Sectional View-Cone Top
Braze or Silver Solder
1/32" Clearance
Scale to be Bent & Brazed on Brackets After Scale is Calibrated
45°
½"
⅛" Brass Screw & Nut
Top View
Top of Pot
¾"
½"
Weld
9/16"
45°
Front Elevation
Side Elevation
Weld
Lug Detail
¼" Pin Ends Split ¼" Deep
1⅝"
Top View
½" USS
Provide Nut & Washer
2⅛"
Drill ¼"
½"D
Lug Bolt Detail

FIG. 2.—Details of Air Meter.

DESCRIPTION OF THE APPARATUS

The construction of the apparatus is shown in more detail in Fig. 2. The cylinder ($\frac{1}{4}$ cu. ft. in volume) is made from standard 8-in. pipe and the bottom from $\frac{1}{4}$-in. plate reinforced with stiffeners. The cone top is also made of $\frac{1}{4}$-in. steel plate, and the column is a section of $1\frac{1}{2}$-in. standard galvanized pipe. A standard waterglass, $9\frac{7}{8}$ in. long, is used for reading

3. Repairs can be readily made in case of accidental breakage.

4. Cleaning of the waterglass can be easily accomplished by the insertion of a test tube brush through the plug opening directly above the glass.

The construction is sufficiently rugged so that no appreciable change in volume occurs under the pressures employed.

TEST PROCEDURE

The container is filled in three layers, each layer being rodded 25 times. Following the filling and rodding of the three layers the container is tapped ten times with a wooden mallet. The upper surface of the concrete is made flush with the top of the cylinder. A 3-in. metal disk is placed at the center of the concrete surface, after which the top section is bolted in place and the apparatus filled with water through a tube and funnel arrangement. Disturbance of the surface of the concrete is prevented by the metal disk.

The water level is adjusted to the zero mark on the scale, following which, pressure is applied by means of a tire pump and the level of the water read. The pressure is released and the scale is read again. The difference between the latter two readings is the percentage of air, as the scale is graduated in terms of percentage of air. The pressure which this Department has used has been 30 psi.

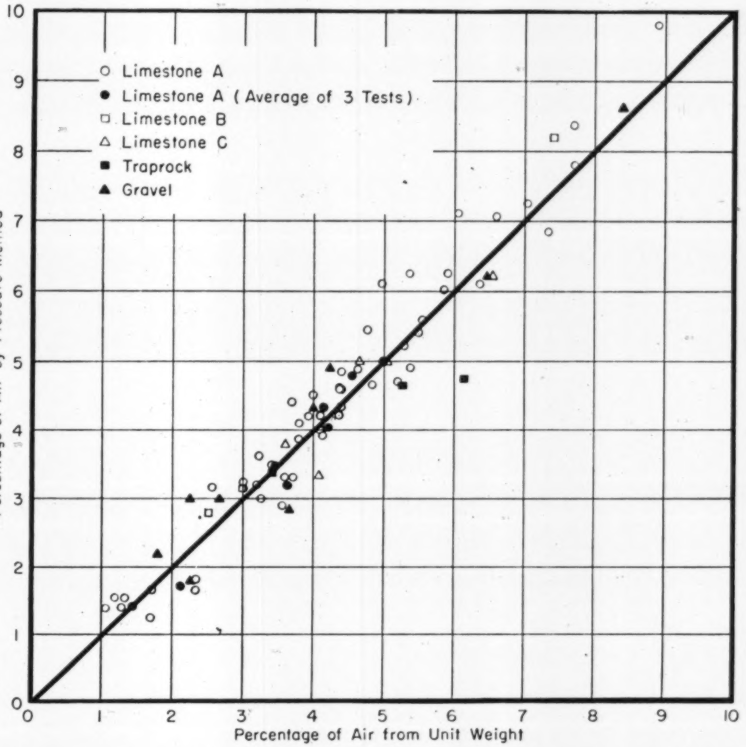

FIG. 3.—Comparison of Air Content Determined by Pressure Method to That From Unit Weight.

PRESSURE METHOD COMPARED WITH THE UNIT WEIGHT METHOD

A series of 75 tests was conducted in which the percentage of air was determined by the unit weight method (A.S.T.M. Method C 138–44)[3] except that the bottom cylinder ($\frac{1}{4}$ cu. ft.) of the pressure apparatus was used. The percentage of air was then determined on the identical sample by the pressure method. Concrete mixtures were tested in which five aggregates of substantially different absorption capacities were used. The slump of the concrete mixtures ranged from $\frac{3}{4}$ to 7 in. The data are plotted in Fig. 3. The pressure method gave higher air contents in 45 of these tests, the average difference being 0.39 per cent. On 30 tests the pressure method gave lower air contents, the average difference being 0.34 per cent. On all tests, the average of the air contents determined by the pressure method was 0.10 per cent higher than the values obtained by the unit weight method. In more than 50 per cent of the tests the difference in results between the two methods was less than 0.30 in percentage points. In view of the many potential errors in the unit weight method, the agreement in test results by the two methods is considered satisfactory.

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[3] Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138 - 44), 1946 Book of A.S.T.M. Standards, Part II, p. 513.

COMPARISON OF DIFFERENT PRESSURES

To determine the effect of the magnitude of the pressure upon the test results, a study was made of concretes in which four different coarse aggregates were used in both an air-dried and a saturated condition. Pressures of 5, 10, and 30 psi., were used. To insure accuracy at the 5 and 10-psi. pressures, a mercury column was used to measure them. Consecutive determinations were made on the same sample of concrete beginning with 5-psi. pressure and ending with the 30-psi. pressure. A scale reading at zero pressure was taken after each of the selected pressures. The air content was determined from the difference between the readings at each applied pressure and its corresponding zero pressure.

TABLE I.—THE EFFECT OF VARIOUS GAGE PRESSURES UPON THE AIR DETERMINATION.

Aggregate	Aggregate Condition	Air Entraining Agent	Concrete Age, min.	Concrete Temperature, deg. Fahr.	Percentage of Air at Gage Pressures of			Average Percentage of Air
					5 psi.	10 psi.	30 psi.	
Gravel	Air dry	Darex	10	70	2.69	2.94	2.85	2.83
Limestone A	Air dry	Darex	10	70	4.32	4.37	4.35	4.35
Limestone C	Air dry	Darex	10	70	4.88	5.04	5.00	4.97
Traprock	Air dry	Darex	10	70	4.88	4.70	4.65	4.74
Gravel	Saturated	Darex	10	70	4.18	4.28	4.35	4.27
Limestone A	Saturated	Darex	10	70	5.02	5.12	5.15	5.10
Limetone C	Saturated	Darex	10	70	6.28	6.22	6.20	6.23
Traprock	Saturated	Darex	10	70	4.46	4.70	4.75	4.64
Limestone C	Saturated	Vinsol	10	70	3.78	3.95	3.80	3.84
Limestone C	Saturated	Vinsol	70	70	3.24	3.36	3.40	3.33
Limestone C	Saturated	Darex	70	70	5.02	5.04	5.00	5.02
Limestone C	Saturated	Vinsol	10	94	3.50	3.36	3.35	3.40
Limestone C	Saturated	Darex	10	87	5.73	6.13	6.15	6.00
Average (13 Tests)					4.46	4.55	4.54	4.52

Absorption Factor

Gravel	1.13
Limestone A	1.95
Limestone C	3.49
Traprock	0.27

The data are shown in Table I. Good agreement is shown between the measured air contents at the three different gage pressures used. Since the aggregates varied in absorption capacity from 0.30 per cent to 3.50 per cent, it appears that the absorption property of an aggregate is not a factor in the determination of the most desirable gage pressure. On the basis of the limited data, it is interesting to note that with one exception the concrete made with the air-dry aggregate contained less air than the corresponding concrete made with a saturated aggregate. Permitting the concrete to stand for 1 hr. longer reduced the air content in the two mixtures tested by 0.5 and 1.2 per cent, respectively. Mixing the concrete at higher temperatures (87 and 94 F.) appears to reduce slightly the air content.

TABLE II.—AIR CONTENT OF COARSE AGGREGATE BY PRESSURE METHOD.

Aggregate	Condition	Moisture, per cent	Percentage of Air at Gage Pressure of		
			5 psi.	10 psi.	30 psi.
Gravel	Air dry	0.16	0.06	0.17	0.22
Limestone A	Air dry	0.30	0.15	0.11	0.30
Limestone C	Air dry	0.67	0.90	1.01	1.10
Traprock	Air dry	0.10	0.01	0.06	0.10
Gravel	Saturated	1.13[a]	0	0.08	0.15
Limestone A	Saturated	1.95[a]	0	0	0.05
Limestone C	Saturated	3.49[a]	0.27	0.42	0.60
Traprock	Saturated	0.27[a]	0.05	0.03	0.06
Water only			0	0.02	0.05

[a] Absorption factor.

TESTS ON THE COARSE AGGREGATE ALONE

Table II shows the values obtained in determinations of the air content of four aggregates in both the air-dry and saturated condition using pressures of 5, 10, and 30 psi. The coarse aggregate, in a quantity equal to that which would have been used in a volume of concrete equal to the volume of the $\frac{1}{4}$-cu. ft. container, was dropped into the water-filled container. The mass was then stirred and the air determination made in the usual manner. The air contents of the air-dry aggregate were comparatively low except for limestone C. In the case of the saturated aggregates, the air contents were practically neglible except again for the highly absorptive limestone C. It would appear that except for highly

TABLE III.—PERMANENT DEPARTURE OF WATER LEVEL FROM ZERO EXPRESSED AS PERCENTAGE OF AIR (AIR-DRIED AGGREGATE).

Aggregate	Absorption Factor	Moisture, per cent	Coarse Aggregate Only at Gage Pressure of		In Concrete Batch at Gage Pressure of	
			10 psi.	30 psi.	10 psi.	30 psi.
Gravel	1.13	0.16	0	0.02	0	0.05
Limestone A	1.95	0.30	0.07	0.12	0.08	0.05
Limestone C	3.49	0.67	0.25	0.25	0.17	0.10
Traprock	0.27	0.10	0	0	0.08	0.10

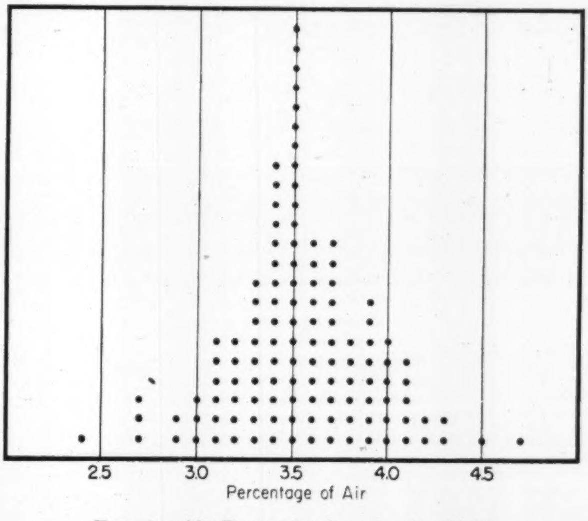

FIG. 4.—Air Determinations in the Field.
Each symbol represents one field test.

absorbent materials, the pores in the aggregates do not seriously affect the determination of air in the concrete.

RETURN OF WATER LEVEL TO ZERO VALUE

The extent of failure of the water level to return to the zero scale reading following the release of the pressure is shown in Table III. Not much difference in magnitude of these values is apparent between the 10 and 30-psi. pressures.

FIELD TESTS FOR AIR CONTENT

Data from field tests on seven paving projects built in 1946 are plotted in Fig. 4, each circle representing one test. The specified range in air content was 2.5 to 5 per cent on two projects and 3.0 to 6.0 per cent on five projects. It may be observed that the average air content was approximately 3.5 per cent and that a comparatively narrow range in test values was obtained.

CALCULATION OF PERCENTAGE OF AIR IN CONCRETE AND THE EFFECT OF ERRORS IN GAGE OR BAROMETRIC PRESSURES

On the basis of the formula below, calculations were made to evaluate the effect of errors in the gage or barometric pressures on the calculated percentage of air determined by applied pressures ranging from 5 to 30 psi. The formula obtained from Stanton Walker is as follows:

$$V_1 = \frac{R(B + h_2 + h_1)\ (B + h_3 + P + h_1)}{(B + h_1)(P - h_2 + h_3)}$$

where:

V_1 = volume of air in the sample of fresh concrete in cubic inches,

R = reduction in the volume of air in the concrete due to the application of pressure in cubic inches,

B = barometric pressure in pounds per square inch,

h_1 = average pressure exerted by the concrete (head of concrete to midheight of measure) in pounds per square inch,

h_2 = head of the water above the concrete, after water has been brought to the mark, in pounds per square inch,

h_3 = head of the water above the concrete after application of pressure in pounds per square inch, and

P = gage pressure in pounds per square inch.

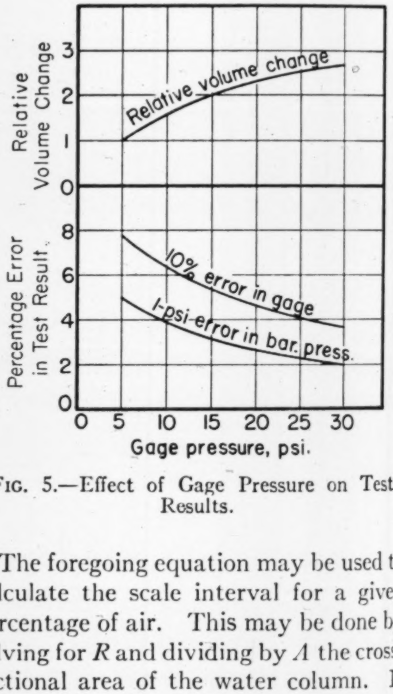

FIG. 5.—Effect of Gage Pressure on Test Results.

The foregoing equation may be used to calculate the scale interval for a given percentage of air. This may be done by solving for R and dividing by A the cross-sectional area of the water column. It will be noted that the term h_3 depends upon the final water level after pressure has been applied. An accurate value of h_3 may be obtained by making a preliminary calculation using an approximate value of h_3 and obtaining the approximate scale interval. The equation may also be solved directly for D, the scale interval in inches, by substituting AD

for R and $h_2 - 0.0361\ D$ for h_3, where 0.0361 is the unit pressure corresponding to a 1-in. head of water. Substitution of these values results in the following quadratic equation in terms of D:

$$V_1 = \frac{AD(B + h_2 + h_1)(B + h_2 + h_1 + P - 0.0361\ D)}{(B + h_1)(P - 0.0361\ D)}$$

This equation must be solved with considerable refinement because the subtracted terms in the final solution are of approximately the same magnitude. The values used in Fig. 5 were obtained from solutions of this equation using the constants for our apparatus. A scale interval of 5.09 in. is obtained for 4 per cent air in a 0.2568-cu. ft. container with the following constants: $B = 14.25$, $P = 30$, $h_1 = 0.38$, $h_2 = 0.54$, all in pounds per square inch and $A = 2.229$ sq. in.

The relative values for volume change R or scale interval D for various gage pressures are shown by the upper curve in Fig. 5. It will be noted that at a gage pressure of 30 psi. the volume change with change in pressure is less than at the lower pressures.

Figure 5 also shows the percentage error in test results for 1 psi. error in the barometric pressure. This corresponds approximately to a difference in altitude of 2000 ft. and is perhaps the maximum variation in barometric pressure which might result from change in weather at a given place. It may be noted that at a gage pressure of 9 psi. the error resulting from an error of 1 psi. in barometer reading is twice that at a gage pressure of 30 psi. It may also be seen in Fig. 5 that the error in test result caused by a 10 per cent error in the gage reading is twice as great at 8 psi. as at 30 psi.

CONCLUSIONS

1. The air-pressure method appears to be an accurate and convenient method for the determination of the air content in concrete.
2. An applied pressure of 30 psi. appears to be suitable for accurate determination of air content.
3. The absorption factor of the aggregate does not seem to be an important factor, except for highly absorptive aggregates.
4. The calculations show that errors in pressure gage reading or barometric pressure are of less effect at the higher applied pressures than at the lower.
5. The apparatus should be of rigid and substantial construction.
6. The apparatus has proven to be more convenient and better adapted for use in the field than the unit weight method.

COMPARISON OF THREE METHODS OF MEASURING AIR ENTRAINMENT IN CONCRETE*

By Alexander Klein,[1] David Pirtz,[1] and C. B. Schweizer[2]

Synopsis

The results of field and laboratory tests on concretes are given as the basis of comparisons of air contents as measured by gravimetric, volumetric, and pressure methods. Following a discussion of the advantages and disadvantages of the methods, brief descriptions of the procedure of testing are given, with certain modifications adopted at the University of California. The test results indicate that the three methods are capable of approximately equal reproducibility; hence, choice of a method for field use could well be made on a combination of such factors as rapidity, simplicity, economy, and the operator's preference.

The three general methods commonly employed to determine the amount of air entrained in fresh concrete are called the gravimetric method, the volumetric or displacement method, and the pressure method. The gravimetric method is indirect, in that the air content is calculated from the weights and specific gravities of the materials and the unit weight of the fresh concrete. Both the volumetric and the pressure methods, with their several modifications, are direct. In the method herein designated as volumetric, the air content is measured by displacing the entrained air with water. In the pressure method, the volume of air is determined by measuring its reduction under applied pressure, in accordance with Boyle's law.

In the investigations at the University of California, reported herein, it was desired that the air content be determined with a degree of accuracy equal to that obtained in the measurement of other properties of fresh and of hardened concretes, but not necessarily higher. It was considered most desirable that the method finally preferred for laboratory or field work be simple and rapid, and especially that it be reproducible within limits implied by current practice in determining the other properties of concrete.

The methods employed, with modifications, were as follows:

1. The A.S.T.M. gravimetric method[3].
2. The A.S.T.M. volumetric method[4].
3. A pressure method identical in principle with, but differing in several matters of equipment and procedure from, that mentioned by Wuerpel and Weiner in 1944 (**1**),[5] and reported in detail by Klein and Walker in 1946 (**2**).

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Research Engineer, Engineering Materials Lab., University of California, Berkeley, Calif.

[2] Lt. Colonel, Corps of Engineers, U. S. Army.

[3] Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138 – 44), 1946 Book of A.S.T.M. Standards, Part II, p. 513.

[4] Tentative Method of Test for Air Content (Volumetric) of Freshly Mixed Concrete (C 173 – 42 T), *Ibid.*, p. 1411.

[5] The boldface numbers in parentheses refer to the references appended to this paper, see p. 885.

ADVANTAGES AND DISADVANTAGES

In appraising the several methods, some general requirements of a suitable test for air content of concrete should be kept in mind. The practical range in air content is of the order of 3 to 5 per cent—at least 3 per cent is required (in the fresh concrete) to develop the desired resistance to freezing and thawing, but more than about 5 per cent of air reduces the strength unduly. In order to be consistent with the accuracy of determination of other properties of concrete, it is estimated that the air content should be determined within, say, 15 per cent of the best obtainable value, and that values obtained by a suitable method be reproducible within, say, 10 per cent of the mean of several values. Within the range of practical air contents, these percentages then correspond to 0.6 and 0.4 per cent respectively of entrained air.

Gravimetric Method:

The data required are the batch weight and the specific gravity of the cement and of the saturated-surface-dry aggregates, the amount of free water in the aggregates, the amount of added water, and the unit weight of the concrete. The accuracy of the method depends on the accuracy of the determination of the properties stated, since the air content is arrived at simply by computation. Wherever the properties of the aggregates and cement remain constant, as in the laboratory, the calculations can be reduced to a simple form,

$$\text{Per cent Entrained Air} = 100 - KF$$

in which F is the unit weight of the concrete and K is a factor varying only with the water-cement ratio. Values of K can be determined for a range of water-cement ratios, a curve plotted, and thereafter values of K taken from the curve whenever desired.

Advantages.—The equipment for this method is that already in use in the laboratory or on any job subject to field control; it is simple and inexpensive. The measurements are those normally made for control purposes; ordinarily no special measurements are required. The calculations are simple and rapid. The physical properties of the cement and of some aggregates usually remain substantially constant for reasonably extended periods. Changes in absorption capacity of normal aggregates do not affect the computed values of air content markedly.

Disadvantages.—For some aggregates, the physical properties vary sufficiently to have considerable effect on the results. Even small variations in specific gravity of the aggregates, if undetected, lead to considerable errors in the computed air content. It is essential that the moisture content of the aggregate be determined individually for each test for air content. The volume of the 0.5-cu. ft. container should be known to within 0.001 cu. ft., and the measurements of unit weight should be reproducible to 0.3 lb. per cu. ft., if the air content is to be determined within 0.2 per cent. The method is hardly practical for small jobs, or at a job receiving ready-mixed concrete. The early shrinkage of the cement-water mixture introduces an undetermined error.

Volumetric Method:

The volumetric method consists in the removal of air from a sample of concrete by mixing it with water in a pycnometer; the volume of air is calculated from the unit weight of a sample containing entrained air and the unit weight of the same sample after it is freed from air. The A.S.T.M. method is a direct method. Several investigators, including the authors, have experimented with various modifications in order to eliminate some of the undesirable features; however, in these modifications certain other undesirable features may have entered. Principally, the modifications have been tried in order to eliminate the objection that air is not completely removed, inasmuch as Pigman (4) has shown that about 0.4 per cent of air is not removed unless a vacuum is used. Other purposes are to avoid or eliminate foam, to reduce the number of weighings and the amount of calculation, and to avoid the use of more than one container. Various methods of rolling the sample in its container and the use of alcohol-water mixtures (to eliminate foam) have been tried. The special methods of rolling usually require special equipment and additional time. The use of isopropanol-water mixtures may cause an undetermined expansion due to precipitation of salts which are less soluble in the mixture than in water alone.

Advantages.—The volumetric method is direct; it involves the observation of a change in volume, which change is apparently proportional to the volume of air. The equipment, though special, is simple and inexpensive. The method is independent of variations in the properties and proportions of the ingredients of the concrete.

Disadvantages.—The chief disadvantages of the volumetric method are its slowness, its complexity, and the uncertainty as to whether the removal of air is reasonably complete. Several vessels are used, and several weighings and other operations are required. The presence of porous aggregate or of excessive foam may cause erratic results. The reproducibility depends markedly on the *degree of reproducibility of placement of concrete in the container*, even in the Indiana modified method (3). For consistent results it is necessary to use standardized containers and uniform methods of placement. Since in this method the pressure head is neglected, the use of containers of different heights results in inconsistencies.

Pressure Method:

The pressure method depends on Boyle's law, applied in its simplest form—the product of the pressure on a gas and its volume are constant. The temperature effect is minor and is generally neglected.

Advantages.—The method is direct, simple, and rapid, and is independent of the proportions, specific gravities, or moisture content of the ingredients. It requires no weighings. The equipment, though special, is inexpensive and simple; it can be constructed by the local plumber from a 12 to 16-qt. pressure cooker, a manometer or a pressure gage, a tire pump, a meter stick, and glass and rubber tubing. The air content can be determined quickly from prepared diagrams; or, for a selected pressure, can be read directly from a specially graduated scale behind the gage glass. For occasional determinations, it could be readily taken to small jobs where none of the usual testing equipment is available.

Disadvantages.—The only significant disadvantage of the pressure method at this time is that it has not been standardized with respect to the method of placing the concrete in the container, the size and shape of the container, and the pressure at which reductions in volume should be measured.

SUMMARY

Considering the advantages and disadvantages just discussed, the authors are in agreement with those investigators, apparently in the majority, who feel that the pressure method is the simplest and most rapid; they feel also that the pressure method offers the greatest possibilities with respect to reproducibility.

It is certainly the most convenient for use in the field.

For laboratory work, as a control and as a reliable check on other methods, it is believed that the gravimetric method (simplified by the use of diagrams) is advantageous. It is best adapted to correlation between various laboratories and between laboratory and field testing.

METHODS OF TESTING

At the University of California, investigations of the properties of air-entrained concretes have been in progress for several years. In connection with these investigations, several methods of determining air entrainment have been used. The data presented herein, however, are limited to those obtained by the three methods described briefly below, as it is believed that these represent about the best practice to be expected in the three general classifications.

Gravimetric Method:

The test procedure was in accordance with A.S.T.M. Method C 138 - 44, except that in the laboratory containers of capacity 0.1 and 0.2 cu. ft. were used in order to afford direct comparison with the other two methods. The use of containers of these volumes instead of the standard size (0.5 cu. ft.) for aggregate of the maximum size employed ($\frac{3}{4}$ in.) would affect the values of unit weight but should have no significant effect on values of reproducibility.

To simplify the calculations, for each of the laboratory concrete mixes used in these tests, various values of water content were plotted against a factor K:

$$K = \frac{100}{V_{g_s}} \left(1 + \frac{W(g_s - 1)}{M + W}\right)$$

where:

V = volume of the unit-weight measure, in milliliters,

g_s = specific gravity of the mixture of cement, sand, and coarse aggregate used in the given concrete mix,

M = weight of cement, sand, and aggregate in the batch, in grams, and

W = amount of water used in the batch, in grams or milliliters.

Thereafter, for each determination of air content, the weight F (in grams) of concrete contained in the unit-weight measure of volume V was determined, and the factor K for the actual water content was taken from the curve. Then,

$$\text{Per cent Entrained Air} = 100 - KF$$

Modified Volumetric Method:

The equipment for this modification of the volumetric method consisted of a unit-weight measure of capacity 0.1 cu. ft., a scale weighing to 0.01 lb., a pycnometer having a volume of 0.3 cu. ft., several small tools and appurtenances, and a supply of *n*-octyl alcohol (primary). The equipment, except the scale, is shown in Fig. 1. The pycnometer was made of aluminum, in order to minimize the weight. Frequent calibrations of the pycnometer revealed no appreciable change in volume.

The unit weight of the air-entrained concrete was determined in accordance with A.S.T.M. Methods C 138 - 44, except that the 0.1-cu. ft. container was employed. The entire contents of the unit-weight measure were then placed in the preweighed pycnometer; the measure was washed out with water and the washings were likewise emptied into the pycnometer. Water was added until the pycnometer was about two-thirds full. Several drops of the normal octyl alcohol were added to prevent foaming. After the pycnometer was rolled in accordance with a fixed procedure, water was added to bring the level nearly

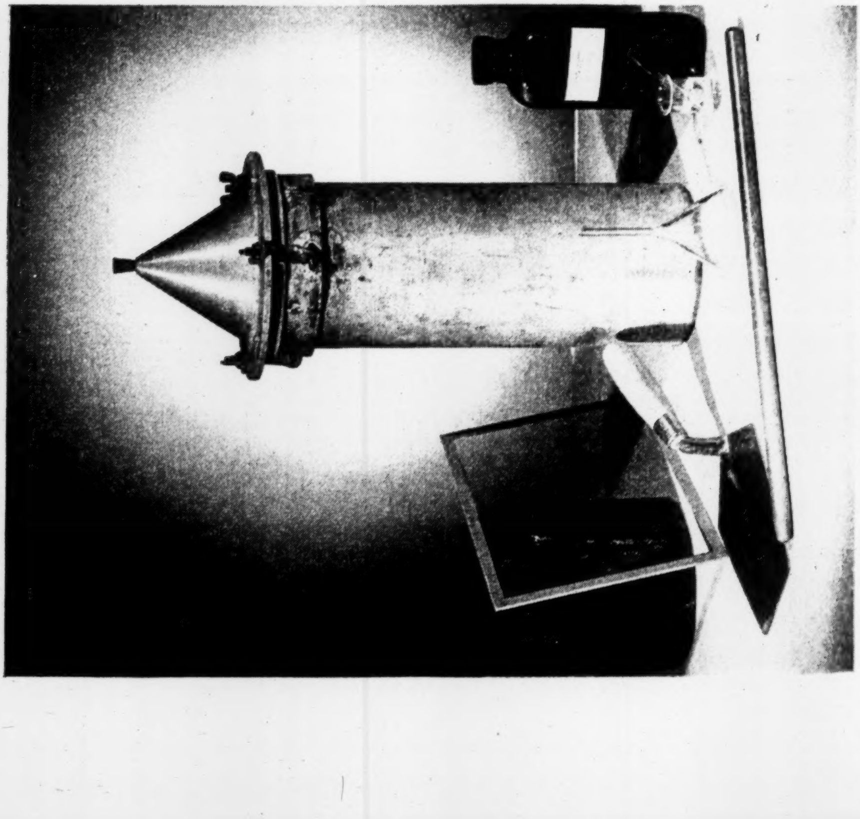

FIG. 1.—Equipment for Authors' Modified Volumetric Method.

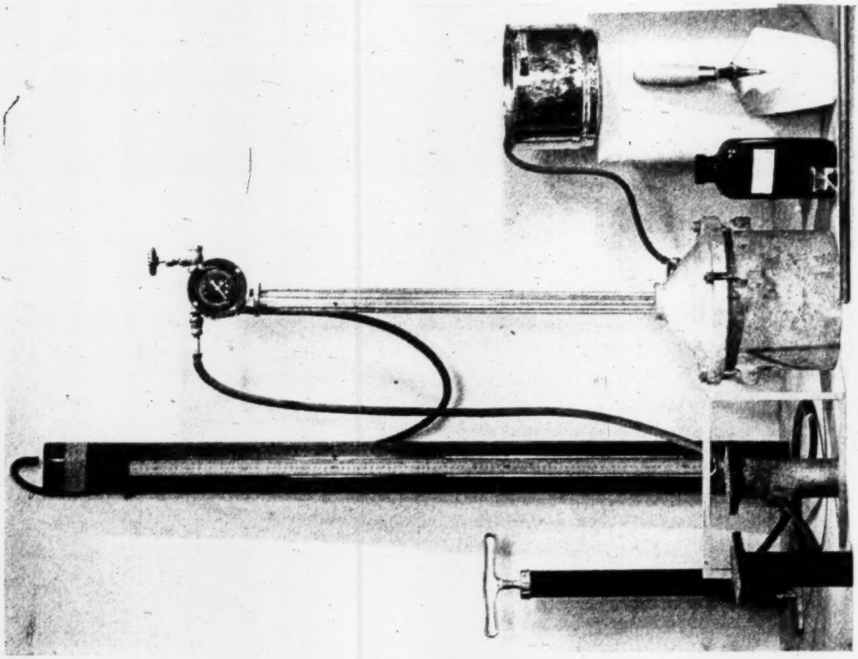

FIG. 2.—Laboratory Pressure Apparatus for Measurement of Air Content of Fresh Concrete.

to the top of the cone. Several additional drops of alcohol were added. Water was then added to fill the pycnometer completely, and the pycnometer with its contents was weighed. The unit weight of the air-free concrete was thus determined, and the original air content computed, in the conventional manner.

Pressure Method:

The apparatus employed by the authors was similar to that employed by other investigators, differing only in detail. One form of the apparatus was constructed for field use and was recently used in the measurement and control of air content of concrete for large precast slabs used as permanent forms and facing for the strengthening of Barker Dam in Colorado. The pressure chamber had a volume of 0.38 cu. ft. A tire pump, coupled to a receiver, was used to produce pressure; and a manometer was used to measure the applied pressure. The assembly was connected with pipe fittings, rubber tubing, and De Kotinsky cement. The apparatus proved to be rugged, and easy to operate. The objectionable features, which were later corrected in the laboratory apparatus, were that the cone was too flat and that the scale behind the gage glass was not movable. At the high elevation where the field apparatus was used (8000 ft. above sea level), trapped air and frothing were not serious problems.

In the laboratory pressure apparatus constructed later (see Fig. 2), the container had a volume of 0.2 cu. ft., the cone was made steeper, a pressure gage was provided in parallel with the manometer, the pressure receiver was omitted, the scale behind the gage glass was made movable, and the gage glass was so mounted as to be rapidly replaced if broken. Calibration containers similar to those described by Menzel (3) were also used to good advantage.

The procedure of testing in the laboratory was as follows: The fresh concrete was placed in the container of the calibrated pressure apparatus in three equal layers, each layer being rodded 25 times with a $\frac{5}{8}$-in. round rod. The side of the container was tapped with the rod until large bubbles ceased to appear on the surface; in no case was it necessary to tap the container more than 15 times. If the concrete appeared to have a slump of less than 3 in., the filled measure was vibrated for 5 sec. on a vibrating table in order to eliminate possible large voids.

The excess concrete was then removed from the slightly overfilled measure by sliding a glass plate back and forth across the top of the measure. (The container with its contents was weighed, in order to provide data for the calculation of unit weight and air content by the gravimetric method.)

The pressure meter was then assembled, and water containing a wetting agent and the anti-foaming agent were added until the level of the water was approximately at a fixed mark on the gage glass. The system was closed, and pressure was applied up to about 20 cm. of mercury. The pressure was released, and the zero of the movable scale was brought to the level of the water in the gage glass. The system was closed again, and pressure was applied in six increments (total pressures 5, 10, 15, 20, 30, and 40 cm. of mercury); at each increment the level of water in the gage glass was read on the scale. The air content for each pressure was read from a prepared curve showing air content against scale reading. (If only one pressure were to be used, as in field testing, the scale could be graduated directly in terms of air content.)

For the apparatus used, and under a pressure of 20 cm. of mercury, an error of 3 mm. in setting and reading the scale would introduce an error of only 0.05 in the calculated percentage of air in concrete containing about 5 per cent of air.

TEST RESULTS

The test results reported herein are selected as being representative of the larger body of field and laboratory tests which have been made over an extended period.

Field Series.—Results of 36 consecutive tests made during the period July 30 to September 17, 1946, at Barker Dam, are summarized in Table I. The concrete had a nominal cement content of 6 sacks per cu. yd. and a nominal slump of 2½ to 3 in.; it contained Type II portland cement and 0 to 1½-in. gravel. Two air-entraining agents were used during the test period, in amounts to bring the air content up to about 4.5 per cent. The pressure method of determining air content was already being employed for speed and simplicity; the gravimetric method was employed during this period as a check on the pressure method. For the gravimetric method, the unit weight of concrete was determined both with the standard 0.50-cu. ft. measure and with the 0.38-cu. ft. container of the pressure vessel.

TABLE I.—UNIT WEIGHT AND AIR CONTENT OF FRESH CONCRETES AS DETERMINED BY GRAVIMETRIC AND PRESSURE METHODS AT BARKER DAM.

Values are average or maximum, as stated, for 36 batches of concrete.

Unit Weight of Concrete, lb. per cu. ft.		
Average (0.50-cu. ft. measure)	146.8	
Average (0.38-cu. ft. measure)	147.4	
Difference (146.8-147.4)	−0.6	
Range in difference between values for individual batches	+0.5 to −1.6	
Average Air Content, per cent of volume of concrete		
Gravimetric method (0.50-cu. ft. measure)	4.6	
Gravimetric method (0.38-cu. ft. measure)	4.2	
Pressure method (0.38-cu. ft. container)	4.3	
Difference in Air Content, per cent of volume of concrete	*Average*	*Maximum*
Gravimetric (0.50-cu. ft. measure) minus gravimetric (0.38-cu ft. measure)	+0.4	+1.0
Pressure minus gravimetric (0.50-cu. ft. measure)	−0.3	−1.4
Pressure minus gravimetric (0.38-cu. ft. measure)	+0.1	−0.9

In Table I, it is seen that the average unit weight of the concrete as determined with the standard 0.50-cu. ft. measure was 0.6 lb. per cu. ft. less than that determined with the 0.38-cu. ft. measure. The individual differences in unit weight ranged from +0.5 to −1.6 lb. per cu. ft., and indicated that the difference in average was not due to error in calibration of either vessel. On the average, the air content calculated from data obtained with the 0.50-cu. ft. measure was 0.4 per cent greater than that for the 0.38-cu. ft. measure; this difference corresponds to about 10 per cent of the air content. The maximum difference (1.0) was about 25 per cent of the air content.

Comparing the air contents as determined by the two methods, it is seen in Table I that the mean value of air content determined by the pressure method (4.3 per cent) was practically the same as the gravimetric value (4.2 per cent) obtained through the use of the same unit-weight measure (0.38 cu. ft.). When the gravimetric value (4.6 per cent) was obtained through the use of the standard 0.50-cu. ft. measure, the difference as between the gravimetric and pressure methods was 0.3, or about 7 per cent of the air content. These relations emphasize the importance of the size of container in correlations between values of air content obtained by different methods.

The consistently low differences as between the pressure method and the gravimetric method evidence the reliability of the simple and rapid pressure method. Perhaps the precision of the pressure-test values is higher than that in ordinary field testing, since the operator (one of the authors) had had considerable previous experience and furthermore knew that comparisons were to be made; but it is not beyond the capability of any careful operator.

Laboratory Series 1.—A series of laboratory tests was made on 36 concretes having a nominal cement content of 6.35 sacks per cu. yd. and a nominal slump of 3 in.; the concrete contained Type I portland cement and 0 to $\frac{3}{4}$-in. gravel. Three air-entraining agents were employed. The operators were graduate students without previous experience in the measurement of air entrainment, but with experience in general laboratory work. For the gravimetric method, the unit weight of concrete was determined through the use of a container of capacity 0.10 cu. ft.

TABLE II.—AIR CONTENT OF FRESH CONCRETES AS DETERMINED IN LABORATORY BY GRAVIMETRIC AND PRESSURE METHODS.

All 36 batches were identical, except that the water content for each group was that which, with the particular air content, would produce the fixed consistency of concrete.

Property	16 Batches (3 to 5 per cent Air)	10 Batches (5 to 7 per cent Air)	10 Batches (7 to 9 per cent Air)
Average Air Content, per cent of volume of concrete			
Gravimetric method (0.10-cu. ft. measure)	3.2	5.5	7.5
Pressure method	4.7	7.0	8.2
Difference (Pressure mius Gravimetric)	+1.5	+1.5	+0.7
Maximum Difference between Values for Individual Batches (Pressure minus Gravimetric)	+2.2	+2.1	+1.8
Average Deviation of Individual Values from Mean of Group			
Gravimetric Method	0.4	0.8	0.7
Pressure Method	0.4	0.9	0.5

The results are given in Table II. For the first group of 16 concretes of relatively low air content (3 to 5 per cent), the average air content determined by the pressure method (4.7 per cent) was about 50 per cent higher than that (3.2 per cent) determined by the gravimetric method. The maximum difference in air content was 2.2 per cent. From a knowledge of the proportions of air-entraining agents in these concretes, it is believed that the better values are those obtained by the pressure method. One factor tending to cause a low gravimetric value was the small size of unit-weight measure; probably the fixed number of 25 strokes with the puddling rod was more effective in the smaller measure.

Similarly, for a second group of 10 concretes of intermediate air content (5 to 7 per cent), the average air content determined by the pressure method (7.0 per cent) was about 25 per cent higher than that (5.5 per cent) obtained by the gravimetric method. The maximum difference in this group was 2.1 per cent of the concrete volume, or about 30 per cent of the air content.

Likewise, for a third group of 10 concretes of relatively high air content (7 to 9 per cent), the average air content determined by the pressure method (8.2 per cent) was about 10 per cent higher than that (7.5 per cent) obtained by the gravimetric method. The maximum difference was 1.8 per cent. In general, the higher the air content the better the agreement between the two methods.

Laboratory Series 2.—Another series of laboratory tests was performed in order to determine the reproducibility of values by each of the three methods. The operators selected to perform these tests were experienced in determination of air content but had not participated in the previous tests reported herein.

Four separate batches of identical concrete were made, and the air content was determined for each batch by four different methods—gravimetric, A.S.T.M. volumetric, modified volumetric, and pressure. Since the tests were made simultaneously, a different operator was employed on each method. A second group of three separate batches was made in like manner, but the composition of the concrete differed in water content, amount of air-entraining agent, and quantity of entrained air. For this group of batches, each operator was employed on a different method from that of the first group.

The results of the tests are given in Table III. Considering first the mean values, it is seen that in general the greatest values of air content were obtained by the pressure method, and the least values by the gravimetric method (in which the 0.10-cu. ft. measure was employed). The volumetric methods were intermediate, and on the average were not far apart.

TABLE III.—REPRODUCIBILITY OF VALUES OF AIR CONTENT AS DETERMINED IN LABORATORY BY GRAVIMETRIC, VOLUMETRIC, AND PRESSURE METHODS.

Mix	Per cent of Agent, by Weight of Cement	Slump, in.	Unit Weight, lb. per cu. ft.[a]	Air Content, per cent			
				Gravimetric[a]	Volumetric[a]		Pressure
					A.S.T.M.	Modified	
No. 1	0.0065	3.9	148.3	4.3	5.0	5.1	5.8
No. 2	0.0065	3.4	148.0	4.4	5.3	6.0	5.3
No. 3	0.0065	3.5	148.5	4.1	5.2	6.0	5.4
No. 4	0.0065	3.0	148.3	4.3	5.1	5.1	5.0
Mean (Mix. Nos. 1 to 4)			148.3	4.3	5.2	5.5	5.4
Average deviation from mean			0.1	0.1	0.1	0.4	0.1
No. 5	0.0022	4.0	151.5	1.9	3.2	3.1	3.4
No. 6	0.0022	3.7	151.4	1.9	3.1	3.1	3.4
No. 7	0.0022	3.8	150.0	2.8	3.8	3.1	3.9
Mean (Mix Nos. 5 to 7)			151.0	2.2	3.4	3.1	3.6
Average deviation from mean			0.6	0.4	0.3	0.0	0.2
Maximum difference in values between tests for corresponding concretes				0.9	0.7	0.9	0.8

[a] 0.10-cu. ft. measure used.

Note: Portland cement ASTM Type I; aggregate 0 to ¾-in. gravel. F.M. = 4.88; nominal cement content 6.35 sacks per cu. yd.; nominal water-cement ratio 0.46 by wt.; nominal slump 3 in.; concrete mixed 3 minutes in a ½-cu. ft. commercial bowl mixer.

Although the reproducibility differed as between the two groups of batches, on the whole it is seen in Table III that there was little difference as among the four methods of testing. However, it is seen that, under these conditions of test, the reproducibility for the gravimetric and the A.S.T.M. volumetric methods was lower at the lower air content (batches 5 to 7) than at the higher air content (batches 1 to 4). For the pressure method, the reproducibility was about the same at the lower air content as at the higher air content, and was relatively high.

For any of the methods, the greatest difference in values of air content as between two batches of the same group was 0.9, or a difference of 0.4 of either from the mean of the two values. This difference from the mean corresponds to about 10 per cent of the air content.

From this limited series of tests it appears that, even with careful workmanship, the reproducibility of an observed value in one additional test by any of the methods is about ± 0.5 per cent of air by volume of concrete. The corresponding total range, 1.0 per cent, is about 25 per cent of the air content. This degree of precision is considerably less than that for measurement of other properties of concrete and less than that stated near the beginning of this paper as being desirable. However, in the present state of knowledge regarding the effect of air content on other properties of concrete, it is difficult to assess the effect of differences in air content less than about 1 per cent.

CHOICE OF METHODS

Since there is no great difference in reproducibility in the methods considered herein, and since it is possible to simplify the procedure and the calculations for the gravimetric and the volumetric methods, it appears that the choice of the method to be used in a given situation will depend largely on the personal preference of the prospective user, with due regard to the availability of the required apparatus and materials. The authors prefer the pressure method because there are no calculations after the apparatus is calibrated and because the likelihood of mistakes is relatively small. It is believed, however, that even in the pressure method it is worthwhile to weigh the concrete in the pressure vessel, to provide data for possible checking by the gravimetric method and for possible correlation studies.

SUMMARY

Previously developed gravimetric, volumetric, and pressure methods of measuring entrained air in concrete, with modifications by the authors, have been compared with respect to simplicity, rapidity, and reproducibility. No quantitative observations of time required for testing were taken, since this factor is affected to a great extent by the operator. It is concluded that the three methods, as modified, are about on a par as to simplicity and reproducibility, but that the pressure method appears to be the most rapid, the simplest, and the surest for either laboratory or field use.

Acknowledgments:

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MEASUREMENT OF AIR CONTENTS OF CONCRETE BY THE PRESSURE METHOD*

By H. W. Russell[1]

Synopsis

Since several sources of error are known to exist in the method of determining air contents of concrete on the basis of weights of samples of known volume, it is desirable that a more satisfactory method be developed. Most of these sources of error do not exist in the pressure method. An apparatus for use in determining air contents of concrete by this method has been constructed, designed for a test pressure of 15 psi., eliminating, as far as possible, all detached accessories

The apparatus essentially provides for the introduction of water above a sample of concrete of known volume to a definite elevation, and the introduction of air above the water until a desired pressure is attained. The subsidence of the water level under the pressure is indicative of the reduction in volume of the air contained in the sample of concrete. The original volume of the air in the sample is determined by the principle of Boyle's law. A graduated scale is provided on which the subsidence of the water level may be read directly in terms of per cent air content when the applied pressure is exactly 15 psi. The air content determined includes not only the entrained air, but also the air held within the pores of the aggregate particles. The proper correction for this may be determined with the same apparatus.

Experimental data show that extreme accuracy in providing test samples of concrete of exact volume is not essential; that the test pressure used, within the limits considered (5 to 30 psi.), is immaterial, and that a high degree of uniformity of results is obtained.

The use of air-entraining cement has been required since the beginning of the 1943 construction season in all concrete pavement constructed under the supervision of the Illinois Division of Highways within the City of Chicago. In August, 1945, the same requirement was extended to apply to all municipalities in Illinois.

Since yield determinations on the basis of weights of a known volume of concrete[2] had been required over a number of years, the air contents of the concrete were determined on the same basis and the results obtained were believed to be fairly reliable. However, this test was not considered entirely satisfactory. Aside from the fact that the performance of it is somewhat burdensome, especially when many tests are desired, several possible sources of error exist which may affect the results obtained.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

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[2] Essentially in accordance with the Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138 - 44), 1946 Book of A.S.T.M. Standards, Part II, p. 513, except that the strike-off operation was performed with a straight-edge.

For accuracy, the test requires knowledge of the exact specific gravities of the materials, the mix proportions, and the amount of mixing water used at the time the test is made, and usually it is impracticable to determine these for the individual batches for which air content determinations are made. Variability of the characteristics and of the condition of the aggregates and inaccuracies in weighing the batches, therefore, introduce error in the results. Accuracy of the results further depends upon field weighings of a container of known volume filled with concrete and struck off even with the rim of the container. Any inaccuracy in performing these operations obviously is reflected in the results of the test, and probably the strike-off operation is the greatest source of error. The Illinois Division of Highways, therefore, has been receptive toward any other method of determining the air content of concrete which showed promise of greater accuracy and simplicity.

Early in 1946, a method utilizing pressure and the principle of Boyle's law in measuring the air content of concrete came to the attention of the Illinois Division of Highways. This method, if it could be made to perform satisfactorily, would not be subject to any error by reason of variability in the materials or batching operations. Also, the effect of error which might exist in the volume of the sample of concrete tested presumably would be relatively small, because the weight of the sample would not enter into the determination of the air content. The pioneering work performed on this method of testing[3] indicated that it was entirely practicable. This was corroborated by information received from the Minnesota Department of Highways.

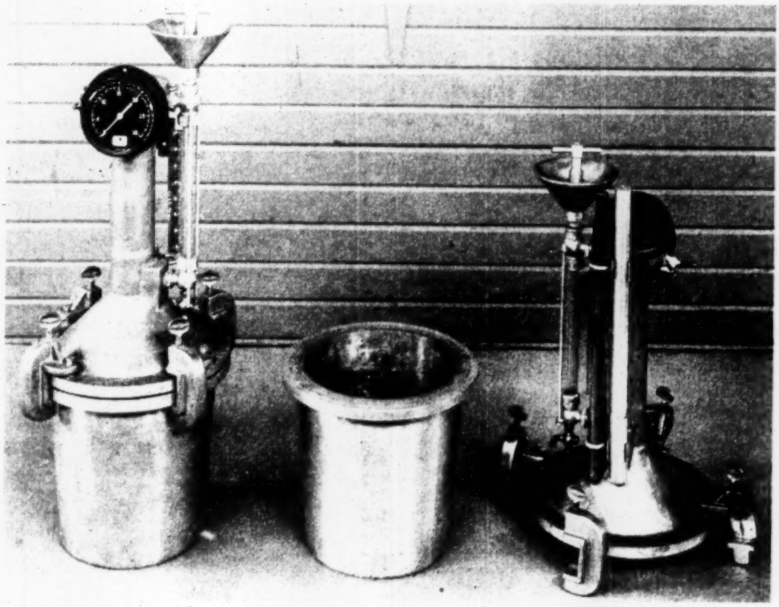

FIG. 1.—Apparatus for Measuring Air Content of Concrete, Assembled and Disconnected.

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[3] W. H. Klein and Stanton Walker, "A Method for Direct Measurement of Entrained Air in Concrete," *Journal*, Am. Concrete Inst., Vol. 17, No. 6, June, 1946.

PRESSURE METHOD—APPARATUS AND TEST PROCEDURE

Early in the summer of 1946, the Illinois Division of Highways built an apparatus substantially in accordance with plans obtained from the Minnesota Department of Highways. Laboratory and field tests thoroughly established the practicability of the method and also indicated that desirable improvements could be made in the apparatus. It seemed particularly important that its weight be reduced to a practicable minimum and that detached accessories be eliminated as far as possible. These and certain minor improvements were incorporated in additional units built in the fall of 1946. The improved apparatus is illustrated in Fig. 1.

The improved apparatus, except for small accessories, is made from a heat-treated magnesium alloy having a tensile strength of about 40,000 psi. It consists of a container of about 0.2-cu. ft. capacity to hold the sample of concrete, a funnel-shaped cover capable of being fastened to the container by a leakage-proof connection, a $\frac{5}{8}$-in. bullet-pointed rod for consolidating the sample of concrete, and a bicycle tire pump for applying air pressure.

The funnel-shaped cover, at its surface of contact with the container, holds a rubber gasket. Spaced around the rim of the cover are four hinged C-clamps for clamping it to the container. Upward from its center extends a standpipe, closed at the upper end.

Near the upper end of the standpipe are a pet cock, a tire stem valve, and a pressure gage. On its side, spaced around its circumference, are two sets of clips to hold the bullet-pointed rod and the bicycle tire pump, respectively, when not in use, and a waterglass for indicating the level of water in the standpipe. A handle will be provided in future units for carrying the assembled apparatus when not in use.

The upper end of the waterglass terminates in a valve and funnel; the lower end in a pet cock for use in adjusting the level of water in the standpipe. Water introduced through the funnel enters the apparatus through the lower waterglass connection and, by means of a tube, is directed against the opposite side of the funnel-shaped cover. This distributes the water and avoids undue disturbance of the surface of the concrete sample. At the side of the waterglass is a scale graduated to show the air content of the sample in per cent when the applied pressure, as indicated by the pressure gage, is 15 psi.

The weight of the apparatus with the container filled with concrete and charged with the proper amount of water is about fifty pounds, or about the same as a similar apparatus made from steel and brass would weigh when empty.

In determining the air content, the container is filled with concrete in three equal lifts, each being rodded 25 times with the bullet-pointed rod, and struck off even with the rim. The contact surfaces are wiped clean and the funnel-shaped cover is clamped on. With the lower pet cock closed and the upper pet cock open, water is introduced until it stands at an initial level indicated by an arrow mark (not shown in Fig. 1) slightly above the zero mark on the graduated scale, any excess water being removed through the lower pet cock. The upper pet cock and the funnel valve are then closed, and air pressure is applied with the bicycle tire pump until the pressure gage reads exactly 15 psi. The subsidence of the water as read on the graduated scale is the percentage of air in the concrete.

The distance between the arrow mark, or initial water level, and the zero mark on the graduated scale corrects for expansion of the apparatus under the applied pressure of 15 psi. This expansion is due chiefly to elongation of the C-clamps under the pressure. The distance between the arrow mark and the zero mark is such that, when the apparatus is filled with water alone to the level of the arrow mark, an applied pressure of 15 psi. will cause a subsidence of the water level exactly to the zero mark.

Since the concrete initially is subjected essentially to the pressure of the atmosphere, the 15-psi. applied pressure roughly doubles the initial pressure, and, in accordance with Boyle's law, the volume of the air in the concrete becomes about half of its original volume. The volume of water forced out of the standpipe, therefore, is roughly equal to half of the volume of air in the concrete. However, in calculating the scale graduations to show percentage of air directly, the sum of the average pressure due to the weight of the concrete in the container, the pressure due to the water column above the concrete, and the average barometric pressure for Illinois, was considered as the initial pressure.

ENTRAINED AIR CONTENT

The air content measured as described includes not only the entrained air but also the air held in the pores of the aggregate particles. To obtain the entrained air content only, it is necessary to correct the reading for the amount of air held within the aggregate particles. This correction may be determined with the same apparatus at the beginning of a job, and the value established may be sufficiently accurate for the duration of the work. The determination is made on separate samples of fine and coarse aggregate, each in the amount which will be contained in a volume of concrete exactly sufficient to fill the container.

With the container partially filled with water, the sample of fine aggregate is added slowly and stirred vigorously to remove the air held between the aggregate particles. This may present some difficulty and requires great care. The container is then filled with water and the cover is clamped on. The further procedure is as described for a sample of concrete. The test is repeated with the sample of coarse aggregate. The sum of the scale readings for the two samples represents the subsidence of the water level due to the air held within the aggregate particles and should be subtracted from the reading obtained for the sample of concrete to obtain the percentage of entrained air. The correction could be determined on the two samples combined, but more difficulty will be encountered in stirring out the air held around the aggregate particles.

CHECK READINGS

After a sample of concrete has been tested and the pressure is released, the water in the standpipe may not return exactly to its original level. However, if the water level is restored by adding water, or if the proper correction is made for the deviation from the initial level, a second application of the pressure will show essentially the same air content as the first. For example, in a series of twelve tests, using a test pressure of 15 psi., the first and second readings both averaged 3.4 per cent, and in a series of 48 tests, using a test pressure of 20 psi., first and second readings both averaged 3.6 per cent. In general, the check between first and second readings has been so close that, in view of the additional manipulations necessary, it is regarded as unnecessary to take check readings.

Failure of the water column to return to the original level after releasing the first application of pressure is believed to be connected in some manner with the aggregates in the concrete. The same has been observed when samples of aggregates are tested to determine the correction for air within the aggregate particles, but the water level has been observed to return satisfactorily in the testing of some sands and mortars. It appears as though a slight amount of water somehow remains within the particles of aggregate after the pressure is released, without actually replacing air.

TEST PRESSURE

In the first apparatus, the test pressure adopted was 30 psi., but it was observed that equally good results were obtained with 15 psi. In the improved units, the test pressure adopted is 15 psi. In a series of 48 tests in which readings were taken, as the pressure was applied, at 5, 10, 15, and 20 psi., the average air contents were determined to be 3.6, 3.6, 3.7, and 3.6 per cent on the basis of the respective pressures. The degree of uniformity of the air contents determined at each pressure, though the apparatus was designed specifically for a test pressure of 15 psi., was approximately the same. There seems to be no reason, therefore, to prefer any one test pressure to another, at least within the range of from 5 to 30 psi. For best performance, however, the apparatus, including the pressure gage, should be designed for the particular pressure adopted. It is of interest that check readings taken at the same time averaged 4.1, 4.0, 3.9, and 3.6 per cent for the respective pressures. Since only the check readings for the 20-psi. pressure agree with the original readings, it appears that any reading taken at a lower pressure than previously applied will be somewhat in error. The further discussion herein applies to air contents determined at the 15-psi. test pressure.

COMPARISON OF COMPACTION AND STRIKE-OFF METHODS

In a series of 18 tests, two units were used in parallel determinations, and the average air content obtained with each was 3.6 per cent. The units were then used in parallel determinations in a second series of 18 tests, in which the samples of concrete were compacted by rodding in one apparatus and by internal vibration in the other. The average air contents obtained were 3.5 per cent for the rodded concrete and 3.6 per cent for the vibrated concrete.

In six of these tests, the compacted samples were weighed, the strike-off process being performed by means of the edge of a trowel; the air contents were calculated from the net weights of the samples. The average air contents obtained were 4.1 per cent for the rodded samples and 3.9 per cent for the vibrated samples, whereas the corresponding values for the same samples, obtained by the pressure method, were 3.6 per cent for both the rodded and the vibrated samples. This difference in results led to further studies of strike-off methods.

In a series of twelve tests, in which the concrete was rodded and struck off with the edge of a trowel, the average air contents obtained were 4.1 per cent, as calculated from the net weights of the samples, and 3.6 per cent by the pressure method. In a similar series of twelve tests in which the strike-off operation was performed with the tamping rod, the corresponding average air contents determined were 2.8 and 3.4 per cent. This shows that accuracy of the strike-off operation is exceedingly important when weighings enter into the determination of air contents, but is not very important in the pressure method. Presumably the sharp edge of the trowel struck off the surface a little too close, whereas the use of the tamping rod on the wide rim of the containers caused the strike-off to be a little too high. In the last-mentioned series of tests, six tests were also made with a container of 0.7-cu. ft. capacity, having a narrow rim. The average computed air content was 3.6 per cent, which compares very well with the air contents obtained by the pressure method.

UNIFORMITY OF TESTING

The summary test values used above were obtained from the results of tests of six concrete mixtures, repeated on six different days, comprising a total of thirty six 0.77-bag batches. The mixtures differed only as to the gradation of the sand, the amount passing the No. 50 sieve being varied from 5 to 40 per cent. The amount of mixing water was 5.4 gal. per bag of cement. The slumps varied approximately from 2 to 4 in. The air-entraining agent was added at the mixer. Two units for air-content determinations were used throughout, but material deviation in procedure was permitted with only one of the units. There resulted, therefore, 36 air-content determinations made under essentially the same condition, except for the variation of the sand gradation, which variation, however, had no discernible effect on the average air contents determined. The only difference noted was that the uniformity of the data for sand gradations showing 10 per cent or less passing the No. 50 sieve was slightly less than for the finer gradations.

In the study of these data, it was decided to disregard one value, 2.1 per cent, the very first determination made in the entire program of tests, which for some unknown reason was entirely out of line with the rest. With this exception, all of the batches showed air contents ranging from 3.2 to 4.2 per cent and averaging 3.6 per cent. Within groups of equivalent sand gradations, only one value deviated more than 0.4 from the average of its group. The standard deviation, whether estimated on the basis of the data within the groups or on the basis of the data as a whole, was found to be 0.22.[4]

This is believed to represent much better uniformity than it is possible to obtain for air-content determinations calculated from the net weights of the samples of concrete. Sufficient data are not available to show the degree of variation on the same basis. However, if it is assumed that the variation in the sand gradation did not affect the air contents, as seems reasonably justifiable, then seven groups of six determinations each on the basis of the net weights are available. The average air content for these groups also was 3.6 per cent, but the standard deviation, as estimated from the group data, was found to be 0.56, indicating a much lower degree of uniformity than obtained by the pressure method.

YIELD DETERMINATIONS

It is of interest that the yield of a batch of concrete may be obtained directly from the air content determined. To do this, it is necessary only to calculate the combined absolute volume of the ingredients in the batch and to increase this absolute volume by the volume of entrained air. This method, since it does not depend upon determining the weight of a known volume of concrete, probably is of greater accuracy than the method now used.[2]

SUMMARY AND CONCLUSIONS

In the discussion presented, the following points appear to be of special interest:

1. The sources of error existing in air-content determinations involving the weights of samples of concrete do not exist or are unimportant in the pressure method.

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[4] The standard deviations shown were obtained by dividing the observed values by the factor c_2 in Table I of Supplement B of the A.S.T.M. Manual on Presentation of Data, and represent estimates of the objective values of the standard deviations.

2. The pressure method determines the total air content. In cases of important porosity of the aggregates, the results should be corrected for air held in the pores of the aggregate particles. This correction is determined by making an air-content determination on the aggregates alone.

3. The pressure method shows satisfactory performance and a high degree of uniformity of results.

4. The test pressure, at least within the range of from 5 to 30 psi., is immaterial, but for best performance the apparatus, including the pressure gage, should be designed for the particular test pressure adopted. The use of a magnesium alloy in constructing the apparatus will do much to lighten the work of performing the test.

5. The test may be used in determining yields of concrete.

ANALYSIS OF METHODS OF MEASURING ENTRAINED AIR IN CONCRETE*

By W. A. Cordon[1] and H. W. Brewer[1]

Synopsis

Determinations of the percentage of air in 45 batches of laboratory mixed concrete by the gravimetric, pressure, and displacement methods show what accuracy might be expected from each method. Errors which might occur in each method are discussed.

TABLE I.—AIR CONTENT OF CONCRETE MIXES.

Mix	Air-Entraining Agent, g. per cu. yd.	Slump, in.	Air, per cent: Pressure Method	Air, per cent: Gravimetric Method	Air, per cent: Indiana Displacement Method	Air, per cent: Bureau of Reclamation Displacement Method
No. 1[a]	50	2.7	6.35	6.09	2.80	4.11
No. 2	40	3.3	6.10	5.53	3.87	4.02
No. 3[a]	30	3.0	5.30	4.40	3.18	4.74
No. 4[a]	35	2.4	4.20	4.42	3.96	3.40
No. 5[a]	35	2.5	3.80	3.66	4.07	3.80
No. 6[a]	35	3.6	4.05	4.11	4.47	4.74
No. 7[a]	37	2.2	3.40	2.68	1.79	3.26
No. 8	40	3.5	4.65	4.77	5.33	4.83
No. 9	40	3.3	5.25	5.63	6.53	3.40
No. 10	40	1.8	4.35	3.95	2.00	2.91
No. 11	40	2.8	5.45	5.16	4.29	3.89
No. 12	40	2.2	5.05	5.06	2.77	3.17
No. 13	40	2.5	5.75	6.06	5.11	5.54
No. 14	40	2.7	5.15	5.26	4.25	5.36
No. 15	40	2.6	3.70	4.22	2.92	3.44
No. 16	40	2.7	3.45	3.46	2.42	3.49
No. 17	40	3.0	4.75	4.84	3.48	3.98
No. 18	40	2.5	4.42	3.92	3.20	3.93
No. 19	40	3.5	4.48	5.27	4.93	4.92
No. 20	40	1.5	3.25	3.08	1.78	1.79
No. 21[a]	45	2.9	5.35	5.82	4.17	3.26
No. 22[a]	45	3.4	5.80	6.27	6.37	4.65
No. 23	40	2.7	5.55	5.26	5.15	3.44
No. 24	40	2.0	3.30	2.97	3.56	2.24
No. 25[a]	43	3.3	5.30	5.42	5.79	3.58
Average air content			4.67	4.65	3.85	3.77
Batch to batch:						
Standard deviation			0.84	0.92	1.29	1.01
Coefficient of variation			18.0	19.8	33.5	26.8

[a] These mixes were not included in computations.

The three most common methods employed in the determination of the amount of air in concrete mixes are:

- (*a*) The gravimetric method (unit weight),
- (*b*) The pressure method (air meter), and
- (*c*) The displacement method (Indiana Highway).

The gravimetric method is similar to that described in A.S.T.M. Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138 – 44),[2] and involves a comparison between the computed unit weight assuming no air, and the measured unit weight of the concrete. The pressure method makes use of the pressure-volume relationship of Boyle's law, and the apparatus used in the tests described in this paper is similar to the Klein-Walker air meter. In the displacement method the entrained air is displaced from a measured volume of concrete by water.

The tests described in this paper were made to investigate the reliability of each of these methods. In order that a comparison might be made of the methods, air determinations were made on duplicate samples from 45 batches of concrete. The results of this comparison are shown in Tables I and II. Two types of displacement methods were included in this comparison: one is currently known as the Indiana Highway method, and the other is similar to that described in A.S.T.M. Tentative Method of Test for Air Content (Volumetric) of Freshly Mixed Concrete (C 173 – 42 T).[3]

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] U. S. Bureau of Reclamation, Denver, Colo.

[2] 1946 Book of A.S.T.M. Standards, Part II, p. 513.

TABLE II.—AIR CONTENT OF CONCRETE MIXES.

Mix	Air-Entraining Agent, g. per cu. yd.	Slump, in.	Air, per cent											
			Gravimetric Method (Unit Weight)				Pressure Method (Air Meter)[a]				Displacement Method (Indiana Highway)			
			Trial 1	Trial 2	Trial 3	Average	Trial 1	Trial 2	Trial 3	Average	Trial 1	Trial 2	Trial 3	Average
No. 26	38	2.2	2.78	2.75	2.82	2.78	3.15	2.55	2.98	2.87	2.18	2.12	1.83	2.04
No. 27	38	2.0	2.26	2.11	2.03	2.13	2.55	2.15	2.15	2.28	2.39	1.72	1.40	1.83
No. 28	38	2.3	1.87	2.13	2.04	2.01	2.15	1.95	1.95	2.01	1.86	1.44	1.59	1.63
No. 29	38	3.4	3.35	3.58	3.98	3.64	3.60	3.20	3.05	3.28	2.34	2.71	2.38	2.48
No. 30	38	3.0	2.01	2.01	2.28	2.10	2.30	2.20	2.25	2.25	1.54	1.47	1.31	1.44
No. 31	38	3.1	4.11	3.52	3.99	3.87	3.85	3.50	3.65	3.66	2.40	1.86	2.66	2.31
No. 32	38	3.0	2.34	2.57	2.60	2.50	2.60	2.50	2.55	2.55	2.14	2.81	1.57	2.17
No. 33	38	2.6	2.00	2.86	2.08	1.98	2.45	2.25	2.35	2.35	1.42	1.38	1.87	1.56
No. 34	38	2.3	2.41	2.25	2.04	2.23	2.55	2.25	2.30	2.37	1.49	0.99	0.92	1.13
No. 35	38	2.3	2.37	2.29	2.35	2.34	2.50	2.15	2.35	2.33	0.95	1.56	1.36	1.29
No. 36	38	2.3	2.40	2.51	2.34	2.42	2.30	2.45	2.25	2.33	0.83	0.85	0.56	0.75
No. 37	38	2.6	3.15	3.42	3.53	3.36	3.05	2.85	3.05	2.98	1.63	1.92	1.95	1.83
No. 38	38	1.6	1.74	1.98	2.08	1.93	2.20	2.15	2.15	2.17	0.97	0.58	1.08	0.88
No. 39	38	2.6	2.70	2.74	....	2.72	2.55	2.50	2.45	2.50	0.24	0.90	2.85	1.33
No. 40	38	3.3	2.91	2.79	2.99	2.90	2.85	2.65	2.60	2.70	1.03	0.44	0.79	0.75
No. 41	38	3.6	3.72	3.94	3.30	3.65	3.55	3.35	3.20	3.37	2.50	1.14	1.82	1.82
No. 42	38	3.6	2.39	2.59	2.71	2.56	2.15	2.35	2.10	2.20	0.97	0.79	1.93	1.23
No. 43	38	3.3	2.51	2.77	2.89	2.72	2.55	2.15	2.10	2.27	1.87	2.75	1.18	1.93
No. 44	38	3.0	3.16	2.92	2.97	3.02	2.85	2.90	2.60	2.78	0.55	0.32	2.58	1.15
No. 45	38	3.3	2.26	2.47	2.55	2.43	2.55	2.50	2.35	2.47	1.22	1.24	1.38	1.28
Average of each method....			2.66				2.59				1.54			
Within batch:														
Standard Deviation........			0.184				0.162				0.498			
Coefficient of Variation.....			6.92				6.25				32.3			
Batch to Batch:														
Standard Deviation......			0.572				0.433				0.491			
Coefficient of Variation (based on average of three mixes)...........			21.5				16.7				31.9			

[a] Aggregate Correction Factor = 0.25 per cent.

DISCUSSION OF RESULTS

From an inspection of the results shown in Tables I and II it appears that equally reliable results can be secured by either the pressure or gravimetric methods when natural aggregates are used. The displacement methods indicate air contents lower than those actually contained in the mixes. The pressure method requires more expensive equipment and requires a greater number of operations than the gravimetric method, but because it requires no computations after calibration and involves only the reading of a pressure gage there are fewer places where large errors can be introduced into the operations if the calibration is accurate. The displacement method is much less reliable than the other two methods as indicated by a much greater variation coefficient shown in Table II. This method also involves considerably more computation than the other two methods. Both the pressure method and the gravimetric method will give improper indication of the air content when good control is not maintained. Table III shows some of the common errors which may creep into these methods if proper precautions are not exercised.

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[3] 1946 Book of A.S.T.M. Standards, Part II, p. 1411.

An inspection of Table I shows what variations might be expected from batch to batch in controlled concrete mixes. This table shows that the indicated air content of approximately 5 per cent may vary from 3 to 6 per cent with no changes being made in the mix parts or mixing procedures. This variation is of consequence only in the laboratory where accurate control is desired. Table II shows the variations in the measured air content in samples of concrete from the same batch. These variations are due to differences in the samples themselves and to the accuracy of the method employed. Assuming no difference in the samples tested, it is indicated that the displacement method is much less reliable than the pressure and gravimetric methods. The displacement method has the comparatively high coefficient of variation of 32 per cent as compared to 6 and 7 per cent for the other two methods.

TABLE III.—ESTIMATED ERRORS IN MEASUREMENT OF ENTRAINED AIR.

Source of Error	Approximate Magnitude of Error for Measurement of 4 per cent Air in 1½-in. Concrete Mix, per cent
Gravimetric (Unit Weight) Method:	
Assume ±0.01 error in determination of specific gravity of aggregate	±0.2
Assume ±0.01 error in water-cement ratio	±0.2
Assume ±5 per cent error in amount of mortar in sample due to segregation	±0.4
Assume ±0.2 error in parts of aggregate	±0.1
Assume an error of ±0.1 lb. weighing unit weight sample	±0.1
Pressure (Air Meter) Method:	
Assume an average variation in barometric pressure of ±0.25 psi	±0.05
Accuracy of reading pressure and water gages ±0.05	±0.1
Assume ±5 per cent error in amount of mortar in sample due to segregation	±0.4
Displacement (Indiana) Method:	
Assume an error of ±0.1 lb. in weighing unit weight sample	±0.1
Assume an error of ±0.1 lb. in weighing volume samples:	
If error accumulates in two weights	±1.6
If error is in one weight only	±0.5
Assume all of the entrained air is not displaced	±0.7
Assume ±5 per cent error in amount of mortar in sample due to segregation	±0.4

With respect to the advantages of the various methods, it appears to the authors that where unit weight determinations are made as a matter of routine mix control, the gravimetric method should be sufficient and adequate for the air determinations in the concrete, and that it should not be necessary to purchase, or use, additional equipment for that purpose. Where adequate control is not maintained and the test is made by inexperienced operators, the air meter is more adaptable for accurate measurement of air.

TEST PROCEDURE

Each batch of concrete consisted of $2\frac{1}{2}$ cu. ft. of material which was mixed in a $3\frac{1}{2}$ S drum-type concrete mixer for a period of 3 min. before dumping into a large pan where it was re-worked with a shovel. For mixes 1 to 25 four men, acting independently, obtained samples from the batch of concrete for air determinations by the gravimetric, pressure, and two displacement methods. For mixes 26 to 45 the procedure was repeated with the exception that three duplicate air determinations were made by the pressure, gravimetric, and the Indiana displacement methods on each batch of concrete using different samples of concrete for each test. This made a total of nine air determinations on each concrete mix. Vinsol-resin air-entraining agent for each batch was taken from a common supply previously mixed and standardized. The prescribed testing procedures listed under References 1, 2, and 3, and methods C 138 – 44[2] and C 173 – 42 T,[3] were followed closely. All data were analyzed in accordance with the A.S.T.M. Manual on Presentation of Data (**4**).

ERRORS IN METHODS TESTED

Table III shows what errors might be expected in the gravimetric method from the use of wrong specific gravity, changes in water-cement ratio, segregation, and mix proportions. It can be seen from this table that a considerable unknown change would have to occur in the mix before any appreciable error would occur in the air determinations, and many of the errors would cancel each other rather than accumulate.

In the case of the pressure method, some of the sources of errors are shown in Table III, and it appears from this table that the largest source of error, where natural aggregates are used, might be due to poor sampling. Segregation and improper sampling are more likely to occur in this method because of the relatively small sample used.

In the displacement methods, it appears that all of the air was not stirred out of the concrete and that, because of this, this method indicates values lower than are actually contained in the mix. As much as 1.5 per cent air was measured by the pressure method after the concrete had been mixed with water, as prescribed in the Indiana test. This will account for the average differences obtained by these two methods.

REFERENCES

(**1**) W. H. Klein and Stanton Walker, "A Method for Direct Measurement of Entrained Air in Concrete," *Proceedings*, Am. Concrete Inst., Vol. 42, June, 1946, p. 657.

(**2**) S. W. Benham, "A Simple Accurate Method for Determining Entrained Air in Fresh Concrete," *Ibid.*, Vol. 42, June, 1946, p. 677.

(**3**) H. F. Gonnerman, "Operation of Pressure Type Entrained Air Indicator of 0.22 cu. ft. Capacity Developed by the Research Laboratories of the Portland Cement Association," Portland Cement Assn. Instructions, April, 1947.

(**4**) Manual on Presentation of Data, Am. Soc. Testing Mats. (1937). (Issued as separate publication.)

THE PYCNOMETER METHOD FOR DETERMINING ENTRAINED AIR IN CONCRETE*

By J. C. Pearson[1]

Synopsis

This paper reviews briefly the development of the pycnometer test for entrained air as a basis for Tentative Method C 173 – 42 T,[2] and the more strictly volumetric method described in the report of Committee C-9 on Concrete and Concrete Aggregates for 1944. Most of the text is devoted to a discussion of errors that are involved in the methods outlined, and suggestions are offered for eliminating the sources of error or for minimizing their effects. In the modifications of the pycnometer that have recently been developed at the Portland Cement Association's laboratories, most of the inherent errors in the method appear to have been overcome, and an improved pycnometer method may therefore be expected to supersede Tentative Method C 173.

The original pycnometer method for entrained air involved a cylindrical pot-type pycnometer of 3 or 4-l. capacity, into which a test sample of concrete of known unit weight was introduced and weighed. From unit weight of concrete and weight of sample, the volume of the latter was obtained, and the volume of water required to fill the pycnometer was then computed as the volume of the pycnometer minus the volume of the sample. However, by nearly filling the pycnometer with water and "washing out" the air entrained in the sample, a larger volume of water was required to fill the pycnometer than the computed volume, the difference being assumed to be the volume of entrained air. This differential volume was determined by means of accurate weighings and the method eventually became the basis of Tentative Method C 173.[2] This is the only method for entrained air now prescribed in A.S.T.M. Standards, aside from the gravimetric or sum-of-absolute-volumes method, C 138–44.[3]

Because of the accurate weighings involved, Method C 173 is not adapted to field work, and under the auspices of Subcommittee VII on Methods and Apparatus for Testing Concrete of Committee C-9, a strictly volumetric method was developed and described in the committee's report for 1944.[4] Without going into too much detail, the volumetric pycnometer consists of three sections, (1) a cylindrical base section of about 0.1-cu. ft. capacity, which serves as a measure for the concrete sample, (2) a cylindrical middle section,

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[1] Director of Research, Lehigh Portland Cement Co., Allentown, Pa.

[2] Tentative Method of Test for Air Content (Volumetric) of Freshly Mixed Concrete (C 173 - 42 T), 1946 Book of A.S.T.M. Standards, Part II, p. 1411.

[3] Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138 - 44), 1946 Book of A.S.T.M. Standards, Part II, p. 513.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 336 (1944).

somewhat larger than the base section and having a conical top, and (3) a graduated glass tube of 250-ml. capacity which is attachable vertically at the top of the cone of the middle section. In operation, the base section is filled full of the concrete to be tested, the latter being rodded in three layers and screeded as it would be for a unit weight determination. The middle section is then attached to the base section, and about 3 l. of water from a measured volume are introduced. The cone is then stoppered and the air thoroughly displaced by turning the pycnometer end for end and rolling it on a table. The graduated tube is next placed in position on top of the cone and water from a measured supply is added until all the scum and foam are collected in the tube. A small measured amount of alcohol is then poured into the tube and stirred with the scum and foam until the latter are completely de-aired. The volume of air in the concrete sample is then the volume of the total added liquid minus the volume of the upper section and that of the liquid in the tube.

In general it may be stated that the pycnometer is capable of giving dependable and reproducible results, provided the work is performed with care and thoroughness. But during the last year or two we have learned that entrained air can be determined with less effort and probably with greater accuracy by a modification of this method or by other methods, and Method C 173 will undoubtedly soon be replaced by an improved method.

The foregoing condensed description of the pycnometer method is perhaps complete enough for the purposes of this symposium, but a brief discussion of the sources and magnitudes of the errors inherent in the method may be helpful in the selection of a better one.

SOURCES OF ERROR

In mixing a concrete batch, a slight shrinkage in volume occurs from the reaction of cement and water. The amount of this shrinkage can be determined approximately by careful determination of the apparent (meaning in this case "fictitious") specific gravity of the cement in slurries of suitable water contents. Tests in our laboratory have indicated that shortly after mixing, a slurry or paste containing 6 gal. per sack will show an apparent specific gravity for the cement which is from 1 to 2 per cent higher than its value as determined in kerosine. Since the cement content of pavement concrete is normally 10 to 12 per cent by absolute volume, the shrinkage in volume of the concrete due to the cement-water reaction is of the order of 0.1 to 0.2 per cent. In the gravimetric test this error appears in the theoretical volume of the concrete on the high side, and the computed percentage of air is therefore too small by this amount in "percentage points." The actual error may be greater than 0.2 per cent if the absolute volume of the cement is computed from an assumed specific gravity of 3.15 when the true gravity is appreciably higher than this, as it is for most type II cements.

The shrinkage in volume of the original concrete that causes an error in the gravimetric result does not appear in the pycnometer test because the latter measures the actual volume of the concrete sample. However, in the washing out process, the sample is diluted with some six to eight times as much water as the sample itself contains, and this is accompanied by further solution of the cement constituents, and further shrinkage that may be even greater than that in the original mix. In our exploratory tests with a type I cement, this apparent shrinkage on dilution in the pycnometer was about 2 per cent of the cement volume, or about 0.2 per cent of the concrete volume. The actual volume of the concrete sample is accordingly smaller upon completion of the test than it was at the start, and the percentage of air as determined is correspondingly too high. The fact that the air content of a given concrete as determined by the gravimetric method is generally about 0.5 per cent lower than that given by the pycnometer method is thus largely, if not entirely, accounted for. It may be pointed out that this secondary shrinkage occurs in the rolling method described by Mr. Menzel, but is practically nonexistent in the Klein-Walker pressure method.

The pycnometer method is subject to three other sources of error that can be largely offset by proper precautions. One of these arises from the use of a compressible gasket which serves to provide a water-tight joint between the two metal sections. If the gasket is compressed to different degrees by the connecting bolts, or if it becomes thinner with use, this may have an appreciable effect on the calibrated volume. An error of 0.01 in. in assumed gasket thickness would cause an error of 0.16 percentage points in an air determination in a 6-in. diameter pycnometer. This error can be avoided by having a metallic bearing between the two sections, such that the gasket is always compressed to a definite thickness. It may be noted that the Klein-Walker pressure meter does not involve such an error, which is another point in favor of this device.

A second error in the pycnometer is the possibility of not displacing all the air from the sample in the washing out process. In our own pycnometer, such an error was found to be about 0.3 percentage points in the case of a sample which seemed to retain its air rather tenaciously. Such an error can be avoided by vacuum treatment or by repeat operations, but the dependence upon these refinements is obviously a real handicap.

A third source of error is the effect of variable water temperatures between the beginning and the end of a test. This would arise only in field determinations made under unfavorable conditions, and a discussion of this error in the 1944 Report of Committee C-9 shows that normally it would be negligible over a considerable range of temperature, and that corrections can be made if necessary by taking into account the known changes in specific volume of water at the observed temperatures.

Finally there is a sampling error of unknown magnitude that may be present when any test sample is taken from a batch of concrete for any purpose. This error arises when the sample is not strictly representative of the batch, that is, when an excess or deficiency of coarse aggregate is present in the sample as compared with the proportion of coarse aggregate in the batch. A recent study of pavement-type concretes in our laboratory has shown that these sampling errors may vary from 0 to 5 per cent or even more, and that the resulting errors in air by the gravimetric method (in this particular concrete) amount to about one-eighth of one per cent air for each per cent excess or deficiency in coarse aggregate. Such errors are insidious, for they give no clue to their presence except in their effect in causing variations in unit weight. Whenever the latter is found to vary by more than 0.2 or 0.3 lb. per cu. ft. in carefully conducted repeat tests, the chances are that sampling errors of appreciable magnitude are present. In our study of such errors, the size of the test sample, in the range from 0.1 to 0.5 cu. ft., seemed to have no effect upon the magnitude of the sampling errors. The reason why these errors were not found to be smaller in the larger samples is not entirely clear, and as Mr. Stanton Walker sometimes says of unexpected test results, this is something to stimulate thinking. This matter is rather important for it has a direct bearing on the size of measures suitable for concrete samples. But fortunately in the pycnometer method, or in other volumetric methods, the errors in air content resulting from sampling errors are considerably smaller than those which appear in the gravimetric method, and should rarely exceed about 0.2 percentage points in the normal range of entrained air.

CONCLUSION

In conclusion, the author's experience has indicated that the pycnometer method is capable of giving satisfactory and dependable measures of entrained air in concrete. However, with the device that has been described, significant errors are likely to be made in air determinations, unless an undue amount of time and effort are spent in overcoming these errors. It is therefore encouraging to learn of the success of the "rolling method" described by Mr. Menzel, which is actually a pycnometer method in principle. In that work he has simplified the apparatus and improved the technique in such manner as to overcome most of the errors that have been described in this paper.

THE OHIO METHOD OF DETERMINING THE AMOUNT OF AIR ENTRAINED IN PORTLAND CEMENT CONCRETE*

By J. F. Barbee[1]

While the Ohio Highway Department has specified air-entrained concrete in its pavements since January, 1943, and in its structures since March, 1945, it was not until March, 1946, that the specifications were altered to require that a definite percentage of air be entrained. At that time, the specifications were changed so as to require not less than 3 nor more than 6 per cent entrained air in all concrete, whether for pavement, structures, or incidental items.

Hook Gage Method

The first method used in Ohio to determine the air content of concrete in the field was the gravimetric method. However, it soon became apparent that some volumetric method of measurement would prove more satisfactory than the gravimetric method, since the latter requires accuracy not generally found in field operations. The Department already had in use on State Highway projects approximately one hundred concrete kits which contained scales of 70-lb. capacity accurate to 0.01 lb., unit-weight measures, tamping rods, scoops, etc. We wanted an accurate method of air determination which required a minimum of additional equipment. Also, we believed that any equipment necessary to the test should be sturdy enough for field use under all conditions. Since the cost of the pressure gage air indicators for general use was for us prohibitive, we decided to use the "hook gage" method, first introduced by the State Highway Department of Indiana. We felt that with the scales available, this method would prove satisfactory if it were possible to stir the entrained air out of the concrete sample. The only additional equipment necessary, then, would be the hook gage itself, which we could make in our machine shop at a cost of about $1.00.

A preliminary investigation was carried on in the laboratory to determine the optimum size of sample and to establish a general procedure for the field. Best results were obtained when the volume of the concrete sample was approximately 40 per cent of the volume of the measure to the hook gage point. With a larger sample, it was difficult to remove all the entrained air, and when a smaller volume was used, it became more difficult to obtain a representative sample. It was also found that extreme care was necessary throughout the entire determination, with special emphasis on the stirring and weighing operations.

Our first adaptation of the hook gage method required four weighings and eight calculations. One calculation—volume of the air-free concrete—was extremely critical, inasmuch as a variation of one point in the third decimal place results in a difference as great as 0.8 or 0.9 in the percentage of indicated air. At that time, however, we had developed no better method, and it was adopted for field use pending further investigation. Concrete inspectors were instructed to use the average of three determinations as the percentage of entrained air in the concrete, and to maintain an indicated air content of 4 to 5 per cent whenever possible.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Engineer, Ohio State Highway Testing and Research Laboratory, Columbus, Ohio.

Next we made a number of comparative tests between the indicated air as determined by the hook gage and the pressure gage on various mixes of concrete, using natural and limestone fine aggregate, and gravel and limestone coarse aggregate. Both pavement and structural mixes were investigated. The comparison of the results on fifteen batches indicated a mean variation of air contents between the two methods of 0.4 per cent, with a maximum variation of 1 per cent. Since examination of the hook gage procedure indicated a maximum variation of 0.9 per cent, it was decided to run another series of tests in which two air determinations would be made by the hook gage method and the average compared with one determination made with the pressure gage.

Using natural sand and gravel aggregates and ordinary and air-entraining cements both with and without an air-entraining additive at the mixer to vary the air content, 32 comparisons were made between the average of two hook gage tests and one pressure gage test (see Fig. 1). The cement factor for all mixes was 6.5 sacks per cubic yard. The air contents indicated by the pressure gage ranged from 1.1 to 7.8 per cent entrained air. The minimum variation between the hook gage and pressure gage determinations in the 32 comparative tests was zero, the maximum 0.9, with a mean variation of 0.36. Analysis of these data indicates that the air content as determined by the average of two hook gage tests would be within 0.5 of the pressure gage result 89 times out of 100, and within 0.2 of the pressure gage result 47 times out of 100. The average air content determined by the hook gage was 4.0 per cent, and by the pressure gage 4.1 per cent.

TEST PROCEDURE—VOLUMETRIC HOOK GAGE METHOD

At this time, H. W. McCoy[2] suggested that perhaps better correlation might be obtained by measuring volumetrically the amount of air entrained. We therefore devised another series of tests in which the air content of the concrete was determined as follows:

1. The weight per cubic foot of the concrete was determined in the usual manner, and the measure emptied and cleaned.
2. The weight per cubic foot of the concrete to the nearest pound was referred to a table which gave the corresponding weight of the sample to be used ($\frac{1}{8}$ cu. ft.).
3. The sample as determined in (2) was weighed into the clean container and jarred vigorously to remove entrapped air.
4. The container was carefully filled with water to the hook gage point.
5. A portion of the water was removed from the container and set aside for future replacement. (The water was removed to reduce the possibility of water being splashed out during the de-airing process.)
6. The concrete was thoroughly stirred to remove the air.
7. The water removed in (5) was replaced, and sufficient water added from a graduated cylinder to bring the water level back to the hook gage point. The air content was then read from the scale on the graduated cylinder, which was graduated directly in percentage of air.

It was found best to wait approxi-

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[2] Highway Bureau, Portland Cement Assn.

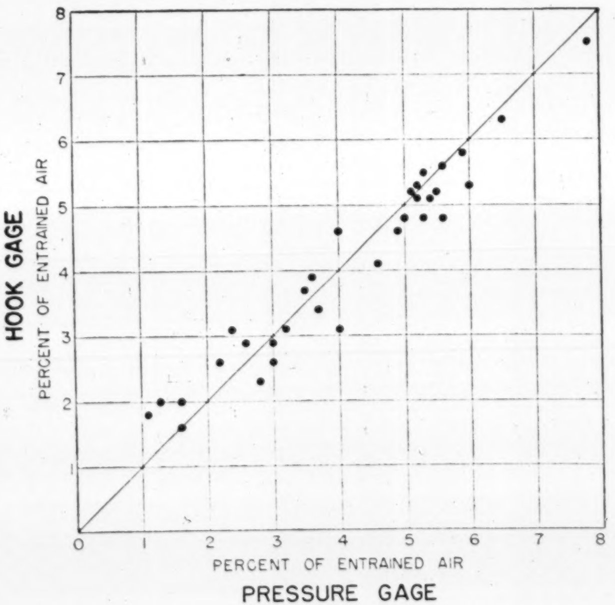

FIG. 1.—Comparison of Percentage of Entrained Air in Portland Cement Concrete as Determined by the Pressure Gage Method and the Hook Gage Method. Pavement mix, 6.5 sacks per cubic yard. Natural sand and gravel aggregates.

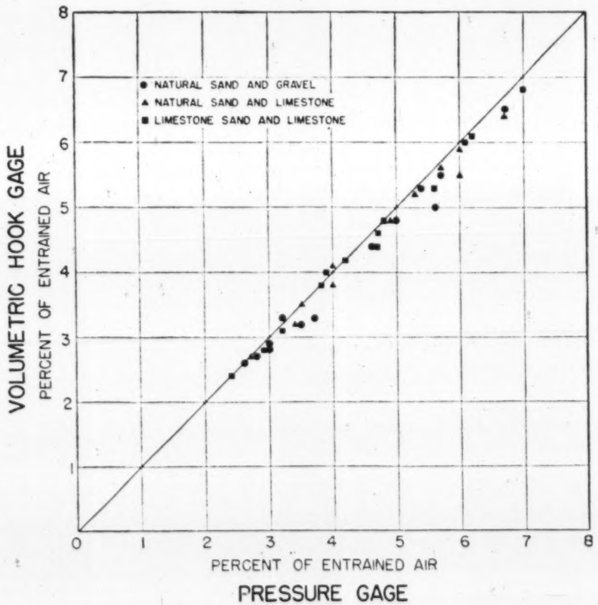

FIG. 2.—Comparison of Percentage of Entrained Air in Portland Cement Concrete as Determined by the Pressure Gage Method and the Volumetric Hook Gage Method. Pavement mix, 6.5 sacks cement per cubic yard.

mately 3 min. after the de-airing was completed before bringing the water level back to the hook gage point, since a slight fall in the level of the water usually occurs. When bringing the water level to the hook gage point, it is advantageous to blow the foam away from the point, and then to touch the point with a finger wet with isopropyl alcohol. The foam is thus dispersed, leaving the point clear.

As an aid to the operator in de-airing the concrete, one end of the tamping rod was flattened to a chisel point approximately 1½ in. wide, and inclined approximately 10 deg., thus making possible a scooping, lifting action which seems to be efficient in removing the entrained air. The lift is obtained by using the edge of the measure as a fulcrum and prying upward.

In order to make sure that all entrained air has been removed by the stirring operation, it is our practice, after the determination has been made, to resume stirring for approximately 2 minutes, followed by a check of the water level.

COMPARATIVE TESTS

The results of determinations by the foregoing method were compared with those obtained by the pressure gage method on the same concrete. Thirty-seven comparisons were made, using concrete made with natural sand and gravel aggregates, natural sand and limestone, and limestone sand with limestone coarse aggregate. Air-entraining cement with and without an air-entraining additive at the mixer was used to provide a variation in the entrained air-content of the concrete. All the concrete was made with a cement factor of 6.5 sacks per cubic yard. Figure 2 shows graphically the relationship between percentage of air as determined by the volumetric hook gage and that determined by the pressure gage on the same concrete. In three instances, a higher air content was indicated by the volumetric hook gage method than by the pressure gage. Seven tests gave identical percentages of air, twelve indicated 0.1 per cent less air by the hook gage than by the pressure gage and eight tests indicated 0.2 per cent less air by the hook gage test than was obtained by the pressure gage. The mean variation between the two methods on the 37 tests was 0.18 per cent. The average air content of the 37 was 4.50 per cent by the pressure gage and 4.36 per cent by the volumetric hook gage. An analysis of these data indicates that in 81 cases out of 100, the result by the volumetric hook gage would be within 0.2 per cent of the pressure gage result and that in 92 cases out of 100 it would be within 0.3 per cent.

CONCLUSIONS

The advantages of the volumetric method over the original hook gage method are manifest: The operation is much simplified, inasmuch as three weighings and seven calculations are eliminated, and the method is much more accurate. Consequently, the volumetric hook gage method has replaced the original adaptation of the hook gage method as a means of air determination in the field on State Highway projects in Ohio.

The graduated cylinder referred to will be made of transparent plastic, glass being considered unsuitable for field use. At present, the cylinders are not available for distribution to the field, so that the water necessary to bring the water level back to the hook gage point after the air has been removed from the concrete is being weighed instead of being measured volumetrically in the cylinder. The results obtained under this improvisation have never in our experience varied more than 0.1 per cent from the determination by the volumetric method.

GENERAL DISCUSSION

MR. BRYANT MATHER.[1]—The Concrete Research Division of the Waterways Experiment Station, Corps of Engineers, and its predecessor, the Central Concrete Laboratory, have been active for a number of years in studies of air entrainment. We have used the gravimetric method as our standard laboratory procedure for determining air content of fresh concrete. Since a large proportion of our work in the Corps of Engineers involves concrete containing aggregate up to 6 in. in size, our particular need has been for a method to determine the air content of such concrete. We have successfully adapted the gravimetric method for this purpose by the use of a calibrated steel container of 3-cu. ft. capacity. While this technique has been found to yield satisfactory results in the laboratory, it is recognized that it would be cumbersome to use in the field. In order to overcome this difficulty a new apparatus is now being constructed for field use. This apparatus consists of a tubular mortar-sampling device and a top which will permit determinations of the air content of a sample of mortar by the pressure principle. Figure 1 shows a diagram of the sampler. It is to be inserted vertically into the concrete, the penetration being made possible by the vibration induced by the vertically acting vibrator mounted at its upper end. As the sampler penetrates the concrete, the coarse aggregate is pushed aside and the mortar enters the sample chamber through the perforated tip. As the mortar rises in the sampler, the air therein is displaced upward and out through the valve at the top of the chamber. When the mortar has filled the tube, the air valve is closed and the sampler withdrawn. Experiments have demonstrated that by means of this technique a sample of mortar may be extracted easily from a concrete mixture containing 6-in. aggregate.

Upon removal from the concrete, the sampler is inverted, the perforated tip removed, the mortar struck off flush with the lower end of the sample chamber, and the base plate attached. The upper end of the sampling device, including the vibrator, is then removed, and the pressure equipment, shown in Fig. 2, is attached. A determination of the air content of the mortar sample may then be made by the pressure method. It will be noted that provision will be made for performing this determination at both increased and decreased pressures. It is felt that experiments may indicate that a particular increased or decreased pressure will provide optimum accuracy and convenience. It is believed that determinations using the method proposed above will be more accurate than those made on the concrete since the range of air contents dealt with will be of the order of 10 to 20 per cent rather than 1 to 5 per cent. It is further believed that no difficulty will be experienced in converting values obtained by this procedure to values of air content of the concrete mixture sampled, since data on the selective

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[1] Corps of Engineers, Waterways Experiment Station, Concrete Research Div., Clinton, Miss.

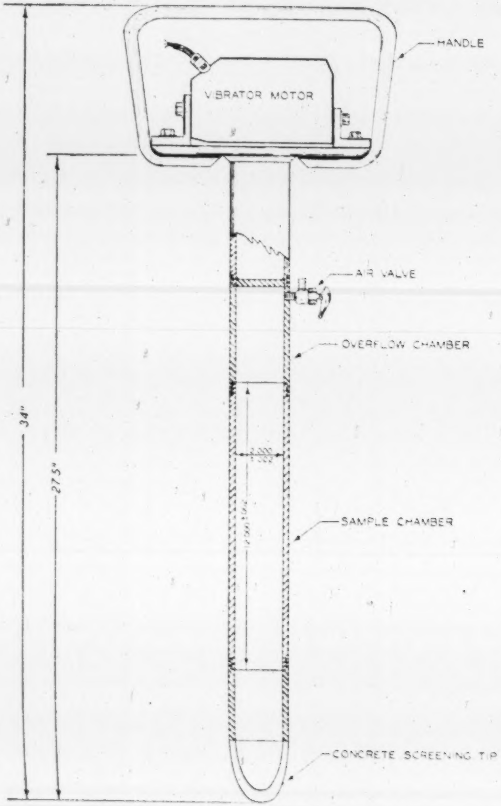
HANDLE
VIBRATOR MOTOR
AIR VALVE
OVERFLOW CHAMBER
SAMPLE CHAMBER
CONCRETE SCREENING TIP
34"
27.5"

FIG. 1.—Mortar Sampling Device.

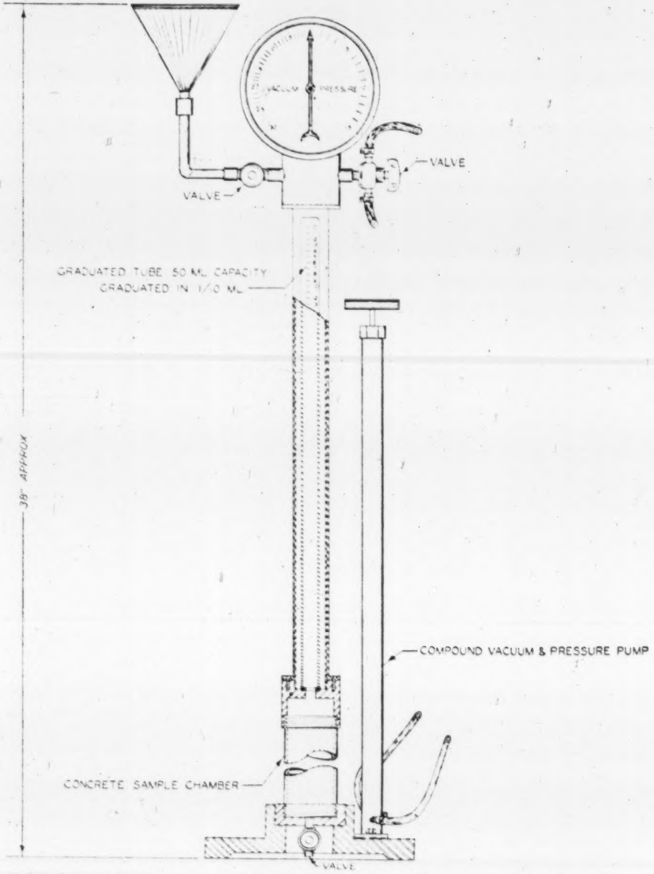
VALVE
VALVE
VACUUM
PRESSURE
GRADUATED TUBE 50 ML CAPACITY
GRADUATED IN 1/10 ML
COMPOUND VACUUM & PRESSURE PUMP
CONCRETE SAMPLE CHAMBER
VALVE
38" APPROX

FIG. 2.—Apparatus for Determining Air Content of Mortar.

screening action of the perforated tip can be easily obtained by a prior test.

I should also like to mention briefly the fact that in our laboratory we have found the gravimetric method to be accurate, rapid, and convenient. We believe that the information on specific gravities and mixture proportions of our laboratory concrete batches is sufficiently accurate so that full reliance may be given to theoretical unit weights calculated therefrom; hence, air contents determined gravimetrically are regarded as satisfactorily accurate. It is frequently necessary in our work to make in rapid succession many batches of concrete containing many different aggregates. For such a program our experience is that air-content data are available on each batch within 1 min. after the weight of the unit-weight measure has been read.

MR. WALTER H. PRICE.[2]—Have you made sufficient tests with your tube sampler to know how reliable this method is? I ask because, without making any tests, I commented unfavorably on a similar method which was submitted for my review. In this method the mortar is slowly drawn into a tube through a screen by a piston which is pulled from a position at the bottom to the top of the tube while the entire apparatus is being slowly turned in the concrete. The "filled" tube is removed from the concrete, the screen unscrewed, and the mortar struck off even with the end of the tube. A cap with a drill rod running through its center and which is sealed against leakage is screwed on to the end of the tube where the screen was. A pressure gage is screwed into an opening provided near the top end of the tube. The drill rod is forced into the mortar until the gage reaches a predetermined pressure, at which point the percentage of air is indicated by the position of the drill rod on a ruled gage.

MR. MATHER.—We have received, I believe, the same plans to which you have made reference and we had considerable doubt regarding the success of the apparatus described. The apparatus which I have described has been demonstrated in our laboratory to be capable of extracting a sample of mortar from concrete. It is my belief that vibration is an essential element in the success of this technique. As far as I know, it has not been demonstrated that a similar sample may be obtained by piston action alone. I might also add, still assuming that the plans to which you refer are the same as those which were submitted to us, that those plans were prepared following a discussion between the author of those plans and Mr. Wuerpel, the former Chief of the Concrete Research Division, in which Mr. Wuerpel explained the apparatus which I have described.

MR. CARL MENZEL.[3]—I should like to ask Mr. Mather the following question: After you have obtained the mortar sample from the bottom of the lift by means of the vibrating tube that you insert into the concrete, and then test the air content of the mortar by the pressure method, what do you do then and how long does it take to obtain the air content of the concrete from the air content of the mortar sample?

MR. MATHER.—We have not as yet made tests on mortar samples extracted with the apparatus which I have described. The complete equipment, illustrated in the figures, has not as yet been completed. We do not anticipate any difficulty in making such tests. In the Corps of Engineers the present specification for air content of concrete requires that "The total calculated air content of that portion of the concrete containing aggregate smaller than the $1\frac{1}{2}$-in. sieve will be 4.5 ± 1.5 per cent of the volume of the concrete." Determinations of the air content of the full mixes may be easily converted into the terms of that specification. Once it has been determined that the perforated tip of the sampling device, which I have described, will exclude aggregate larger than a given size, it is believed that by similar arithmetic processes the air content of the entire mix or of that portion of the mix containing aggregate smaller than $1\frac{1}{2}$ in. may be calculated.

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[2] Materials Engineer, U. S. Bureau of Reclamation, Denver, Colo.

[3] Senior Research Engineer, Portland Cement Assn., Chicago, Ill.

MR. MENZEL.—How do you determine the solids in this little mortar sample?—that is really what you have to do before you know very much about that sample. You cannot assume that the solids are screened out uniformly by the little tube that you insert in the concrete. If you have a way of determining the cement, sand, and water in the mortar, then you have the information that you need to calculate the air content of the concrete. But how are you going to do this quickly and accurately?

I have thought a lot about the subject of testing small mortar samples and have concluded that what we really are interested in is the air content of the paste, and when we have this information we should not have to be concerned about the air content of the concrete or whether the mix contained aggregate as large as 6 in. in diameter, or only sand grains. The paste basis should be good enough for most purposes, provided a way is found to determine the composition of the mortar sample which Mr. Mather withdraws from the concrete.

MR. M. A. SWAYZE.[4]—Our research laboratory has been quite interested in all of the methods of determining entrained air, not so much in the laboratory, but in the field. It was not until I read Mr. Pearson's very fine paper, in which he subjects his own method to critical analysis, and until I read his heading of one section of it, "Sources of Error," that I was prompted to study differences in the other test methods to see where sources of disagreement between them might occur.

As far as field methods are concerned, I think we can dismiss the gravimetric method from consideration for this reason: we very, very seldom know exactly how much water we have in a sample of concrete. We may weigh stone, we may weigh sand, and we may measure the water, but no one knows accurately from batch to batch just how much water is in the aggregates as they are weighed. So that method used in field work is open to serious doubts. Similarly, the Indiana Method will work well in the laboratory, but in the field it is apt to show quite wide variations from a cause that is not apparent upon the surface; that is, when concrete is rodded, especially the type that is used in highway work, and especially where angular crushed stone is used for coarse aggregate, the coarse aggregate is very likely to be rodded down toward the bottom of the measure. Then when the top half is dumped out more mortar is dumped proportionately than coarse aggregate and consequently, the air being in the mortar, the results will be low.

Comparing the pressure method and the rolling or pycnometer methods, there are two discrepancies between these that have not been brought out and yet which are fundamental. If we go over the method of calculating air by the pressure method, we find that we bring the air back to its pressure under one atmosphere. In the rolling method or pycnometer test, the air as we determine it is under the additional pressure of half the height of the concrete in the test cylinder plus the height of water which is above it. There is some difference in the volume of air under simple atmospheric conditions or under the slight additional pressure to which it is subjected in the test measures. So there is a discrepancy there, with the pressure method of calculation giving slightly higher results.

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[4] Director of Research, Lone Star Cement Corp., New York, N. Y.

Then a second point, regarding the pressure method alone. Mr. Menzel mentioned it and I am sure that everyone who has run pressure tests has found the same thing to be true. After the water column is brought to the zero point, pressure is applied, the depression of the water column is measured, and then the pressure is released, bringing it back to atmospheric, but the water column does not return to zero. This lag in the water column is deducted from the gross quantity of air indicated.

Mr. Menzel and others have explained that this failure of the water column to return to zero is due to the absorption of air in the difficultly permeable pores of the aggregate. That has been a little hard to accept, but there is a much easier explanation. Air is soluble in water, and we can assume, I believe, that after the period of mixing has gone on, the water in the sample of concrete is saturated with air at atmospheric pressure. The air content of the concrete is pretty well dispersed in tiny bubbles. It has a relatively high surface area. When you subject an air-water mixture under such conditions to an additional half atmosphere pressure, as in Mr. Menzel's modification of the Klein-Walker apparatus, or in others where pressure is even greater, you should find quite a rapid solution of air in the mixing water in the container. Once the air has gone into solution, it does not come out very rapidly. Mr. Menzel has given 20 to 30 min. for the time for the columns to return to zero. Therefore, I question whether we should, in the determination of air by the pressure method, subtract this drop in the water column which occurs when we first place the pressure on the apparatus. This means, of course, where there is now only a very slight increase in air by the pressure method over that indicated by the rolling method, etc., due to the differences in the pressure on the air as we determine it in one or the other method, we will have somewhat greater difference between the two methods if we can demonstrate that this loss in volume of air, as we measure it, is due to solution of air in mixing water. However, the recommended range given by the Portland Cement Assn. for air entraining concrete of 3 to 5 or 6 per cent, does not need to be changed if we change our method of determining air by the pressure method. The minimum of three per cent is very amply above any danger line. If you go over the original reports in the Am. Concrete Inst. *Journal*, June, 1944, where those recommendations were first given, you will find you do not start on the up curve of high losses, for either modulus of rupture, sonic modulus, or weight, or increases in length due to action of freezing and thawing, until you get well below 2½ per cent. I suggest that we look into this matter of the small difference between the original zero point and the return of water to below the zero point and determine whether it is really absorption of air by the aggregate, or whether it is simply solution of air in the water in the sample.

MR. R. A. BURMEISTER.[5]—I should like to offer a few words in defense of the gravimetric method for testing for air content. In our laboratory we use both the gravimetric method and the pressure method, and we find it necessary to use both for good control of our city paving projects and other concrete projects.

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[5] Materials Engineer, City of Milwaukee, Testing Laboratory, Milwaukee, Wis.

It seems that criticisms of the gravimetric method on the basis of accuracy and speed tend to overlook the fact that the gravimetric method gives valuable data such as yield of concrete, specific gravity, moisture content, and absorption of aggregates—all of which should be taken for a well-controlled job. I have been watching pavement jobs on state, city, and airport work, and notice that recently it has apparently become fashionable to make air-content tests (mostly by the pressure method) to the virtual exclusion of all other concrete tests. A few years ago the fashion was to make beam tests. It seems to me that the type and number of tests made on concrete work should depend entirely on their practical field value and not on a hair-splitting comparison of accuracy. The gravimetric method may not produce air-content determinations as reproducible as those obtained by the pressure method but it is a good test nevertheless. From the standpoint of field control the two methods should be regarded as complementary—not as competitive.

Incidentally, it is our custom to take aggregate samples for specific gravity, moisture, and absorption tests out of the very batch that we use as a subject for the gravimetric test; in this manner we avoid a common source of error—nonrepresentative aggregate samples.

A further refinement we sometimes use in the gravimetric method is to correct the observed air content of the concrete by adjusting for the absolute volume of mortar in the measure. For this correction a gravel "wash out" test is made.

MR. F. S. MERRITT.[6]—It seems to me that the object of these tests is to measure the amount of entrained air which is supposed to have a beneficial effect on the concrete. Now, I wonder whether in the ordinary mixing of concrete, there is not a certain amount of ordinary air entrapped, which may prove detrimental as far as the characteristics of the final concrete are concerned.

In all these tests, it appears that we measure the total amount of air in the concrete without differentiating between the minute bubbles of air and the larger volumes of entrapped air. Do any of these tests measure the actual entrained air, as contrasted with the entrapped air, so as to indicate the net result to be expected in the properties of the concrete due to the presence of both types of air pockets?

MR. MATHER.—The Concrete Research Division has attempted to distinguish between entrained and entrapped air by two different procedures, one indirect and one direct. Indirectly we have made comparable concrete mixtures with and without the use of air-entraining admixtures, the differences between the determined air contents being regarded as the amount of entrained air as compared with the amount of entrapped air. The direct method involves the study by microscope methods of polished surfaces of hardened concrete. By this technique the actual air voids themselves may be observed, measured, and classified with respect to size, the larger ones being regarded as the result of entrapped air and the smaller ones being regarded as resulting from the action of the air-entraining agent.

MR. H. W. RUSSELL (*author's comments by letter.*)—Divergent views have been expressed as to whether the difference in the water levels in the pressure method before application of pressure and after its release should be considered as indicative of air in the concrete. My own view, apparently shared by Mr. Swayze, is that the subsidence of the water level under the applied pressure is indicative of the air content of the concrete, whereas Mr. Menzel and Messrs. Swanberg and Thomas consider the recovery of the water level upon release of the pressure as indicative of the air content.

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During the time elapsed since the preparation of my paper, we have made some tests to try to find out just what happens to the mortar and coarse aggregate components of concrete when pressure is applied and released. The action of mortar and coarse aggregate particles were observed separately when tested under a 10-psi. pressure in a glass container, effectively screened to prevent injury to the observer in the case of breakage of the glass. Samples of mortar, during application and release of pressure, were observed to compress and then expand with no more than an occasional air bubble escaping into the water. In some similar tests made later, there was evidence of some air escaping upon release of pressure in the form of very minute bubbles, visible only under strong light. It seems fairly certain that a small amount of air escapes from the mortar upon release of the pressure, no doubt an insignificant quantity as compared to the total amount therein. In the case of coarse aggregate particles, no air was observed to escape while the pressure was being applied or maintained, but immediately upon release of the pressure, plainly visible air bubbles escaped from the coarse aggregate particles. In fact, it was found that a considerable degree of absorption of water by porous objects could be obtained by several successive applications and releases of pressure.

In another test, a 4 by 8-in. concrete cylinder, in oven dry condition, was tested under a 15-psi. pressure maintained for 7 hr. 20 min. by connecting the apparatus to an air line through a reduction valve set for this pressure. The water level subsided fairly rapidly at first, reaching a nearly constant level in about 3 hr., at which time the reading was 3.9. At the end of the test the reading was 4.2 and, upon release of the pressure, the water level recovered to 4.1. This shows that practically all of the water forced into the concrete remained therein, amounting to nearly 7 cu. in., not including the amount of probably more than 1 cu. in. absorbed before the pressure could be applied. When the cover of the apparatus was removed, the specimen was observed to emit streams of air, and 3 hr. 30 min. later, when last observed, it was still emitting air. Tests of shorter duration on additional specimens showed comparable results.

In view of these observations, we have concluded, in the case of determinations of the aggregate correction, that only the reading under the first application of pressure indicates the true correction. After release of the pressure the situation has changed. Some air has escaped from the aggregate particles, and some air remains under pressure within the aggregate particles for some time, as in the case of the concrete cylinder described above. Some water has entered the aggregate particles, preventing the water level from returning to its initial position after release of the pressure.

We have further concluded, in the case of air content determinations of concrete, that the situation as far as the aggregate is concerned is about the same as described above, except that at least most of the air escaping from the aggregate particles probably remains in the mortar. The amount of water entering the aggregate particles is greater than the amount of air expelled from them (since some air under pressure remains), and for this reason, and because there no doubt is a slight loss of air from the mortar upon release of the pressure, the water does not return to the original level. It would seem, in this case also, that only the reading under the first application of pressure would show strictly the correct air content. However, if the aggregate is only slightly porous, and if only a small amount of air is lost from the mortar, a second application of pressure, the original water level having been previously restored, no doubt would show essentially the same result as the first.

It is my belief that these tests and the discussion presented herein support my view that the total subsidence of the water level, excepting that which may be due to expansion of the apparatus, represents air content.

The testing procedures described by the various authors, with respect to consolidating the sample, more or less follow the procedure specified in the gravimetric method. Filling the container in three layers and rodding each 25 times is generally adhered to, though the size of the container in most cases is less than half of that specified in the gravimetric method. Further work in consolidating the sample ranges from vigorous tapping of the external surface of the container, as specified in the gravimetric method, to no tapping at all.

Tapping of the external surface of the container causes air bubbles to rise and escape from the concrete, which may be observed by placing a small sample of mortar in a glass jar, adding sufficient water to make visible the escape of air bubbles, and tapping the jar lightly on the side. Rodding also causes air to escape, as does vibration. Internal vibration exactly sufficient to consolidate the concrete has been observed to show essentially the same air content as rodding. However, internal vibration after consolidation by rodding removes additional air, and concrete having been consolidated by internal vibration, then scooped into the container and consolidated by rodding will show reduced air content. Indications from a few tests are that the additional rodding or vibration removes in the vicinity of 0.75 per cent air, depending somewhat on the original air content of the concrete. The amount of work expended in consolidating the sample, therefore, has a bearing upon the air content determined.

It is my opinion that the amount of work expended should be no more than just sufficient for consolidating the concrete, and that vigorous tapping of the external surface of the container, as specified in the gravimetric method, may release an undue amount of air from the concrete.

Whether it is most expedient to adopt a definite test pressure and determine the scale graduation interval to show percentage of air, or to adopt a definite scale graduation interval to show percentage of air and determine the test pressure to be applied, is undoubtedly a matter of individual preference. However, I do not believe that the test pressure used should be too low, because, as pointed out by Messrs. Swanberg and Thomas, error in the applied pressure has a greater influence on the indicated air content at low test pressures than is the case at higher test pressures.

Correction for significant difference in elevation above sea level from that for which the apparatus is calibrated is necessary. We have given some consideration to the use of a test pressure exactly equal to the atmospheric pressure. The test pressure, at any elevation, without significant error, would be the nominal atmospheric pressure for that elevation. This plan, however, was considered entirely unnecessary for Illinois, but I believe it would have considerable merit for mountainous states and in the case of country-wide standardization.

A calibration cylinder, as described by Mr. Menzel, for occasional checking of the accuracy of the pressure gage should be available. However, it has been our experience that it is very difficult to avoid loss of air from a cylinder of this type. We have obtained very satisfactory results with a cylinder having a constricted opening, somewhat in the shape of an inverted bottle, for which the loss of air is too slight to be noticeable.

THE EFFECT OF SAMPLING ERRORS ON UNIT WEIGHT AND AIR DETERMINATIONS IN CONCRETE*

By J. C. Pearson[1] and S. B. Helms[1]

Synopsis

If a test sample from a batch of freshly mixed concrete is taken for analysis, the chances are that the ratio of coarse aggregate to mortar in the sample will not be exactly the same as that in the batch. The excess or deficiency of coarse aggregate in a nonrepresentative sample is arbitrarily called the sampling error, and a method for evaluating this error and its effect on computed unit weight and entrained air is given in detail.

Data from tests of 30 batches of pavement-type concrete show that accurate unit weights and air contents can be derived from small test samples when corrections are made for sampling errors, thus providing a means of avoiding errors from loss of mortar in the mixer. Other changes and possible improvements in testing procedure are suggested on the basis of this study.

In various laboratory studies of methods of determining the air content of concrete, the authors have been concerned over the recurrence of rather large discrepancies in unit weight determinations made from samples of a given concrete, not only in measures of different volumes, but also in repeat determinations in a given measure. These larger discrepancies were first noticed in low-slump, pavement-type concrete and seemed to be of similar magnitude when measures of 0.1- and 0.5- cu. ft. capacity were used. This led to the assumption that nonagreement in unit weights was due to nonuniform consolidation, but after special attention had been given to this operation, without any distinct improvement in results, the conclusion was reached that the concrete samples themselves might be lacking in uniformity. While standard A.S.T.M. specifications pertaining to concrete recognize a need for larger measures with increasing maximum size of aggregate, presumably for the purpose of minimizing sampling errors, the authors are unaware of any definite basis for the selection of the sizes of measures specified, and it therefore seemed worthwhile to investigate the occurrence and effects of nonrepresentative samples on the accuracy of unit weights and of results derived from them.

In the following discussion it will be convenient to refer to an excess or deficiency of coarse aggregate in a sample of concrete, as compared with the proportion of coarse aggregate in the batch from which it is taken, as a *sampling error*. It will be seen from the test data presented herewith that sampling errors of appreciable magnitude are practically always present, often negligible, but frequently affecting the unit weight and computed air content by significant amounts.

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DERIVATION OF EQUATIONS FOR SAMPLING ERRORS AND THEIR EFFECTS

If the weight and volume of a batch of concrete are W_B and V_B, respectively, and the weight of a sample of concrete in a measure of volume V_M is W_M, the volume of the batch is ordinarily taken as $V_B = \frac{W_B}{W_M} \cdot V_M$. If the sample is not exactly representative of the batch, W_M will be in error, and V_B will consequently be in error. Now if it be assumed that the nonrepresentative character of the sample is due to excess or deficiency of coarse aggregate, with a corresponding deficiency or excess of mortar, and if it be further assumed that the mortar in the sample is representative of that in the batch, the so-called sampling error can be quantitatively determined and its effect on unit weight and computed air-content can be calculated. To do this it is necessary to recover the coarse aggregate from the weighed sample of concrete in the measure, and determine its weight. (Throughout this discussion the weight of aggregate will mean its weight in saturated, surface-dry condition, unless otherwise stated.)

Let $W_s + \Delta W_s$ be the weight of the recovered coarse aggregate, where W_s is the weight of the coarse aggregate that would be present in a truly representative sample, and ΔW_s is the weight of the excess (or deficiency, when negative) of coarse aggregate. If ΔW_s is positive it would displace an equal volume of mortar, and the weight of the concrete sample would therefore be too high by ΔW_s less the weight of an equal volume of mortar. This volume is $\Delta W_s/62.3\ g_s$, where g_s is the specific gravity of the coarse aggregate in saturated, surface-dry condition, and the weight of the deficiency in mortar is $g_m \times \Delta W_s/g_s$, where g_m is the specific gravity of the mortar. The error in the weight of the sample is therefore $\Delta W_s - g_m \times \Delta W_s/g_s$. Hence the equation

$$W_M = W_T + \Delta W_s \left(1 - \frac{g_m}{g_s}\right) \ldots (1)$$

where W_T is the weight of the concrete sample when it is truly representative of the batch, and the other terms are as previously defined. For any given concrete, the ratio g_m/g_s is fixed, and Eq. 1 may be written

$$W_M - W_T = K\,\Delta W_s \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

Equation 2 shows that the error in the weight of the sample is directly proportional to the sampling error. The ratio g_m/g_s is equal to the weight of the mortar divided by the weight of an equal volume of coarse aggregate; hence

$$K = 1 - \frac{g_m}{g_s} =$$

$$1 - \frac{W_M - (W_s + \Delta W_s)}{62.3\ g_s \cdot V_M - (W_s + \Delta W_s)} \ldots (3)$$

The value of ΔW_s can be expressed in a single equation, but it is simpler to derive it in a series of steps. We first need to know the true volume of the batch, which is obtained by dividing the weight of mortar in the batch by the weight of mortar in the sample, and multiplying the quotient by the volume of mortar in the sample; this gives the volume of mortar in the batch, which, together with the absolute volume of the coarse aggregate in the batch, constitutes the true volume of the batch. The steps in this derivation are as follows:

Weight of mortar in batch = weight of batch (W_B) − weight of coarse aggregate in batch (W_{CA}) .... (A)

Weight of mortar in sample = weight of sample (W_M) − weight of recovered coarse aggregate ($W_s + \Delta W_s$) .... (B)

Volume of mortar in sample = volume of measure (V_M) − volume of recovered coarse aggregate $\left(\frac{(W_s + \Delta W_s)}{62.3\ g_s}\right)$ ...... (C)

$$\text{Volume of mortar in batch} = \frac{(A)}{(B)} \cdot (C) \ldots\ldots \quad \text{(D)}$$

$$\text{True volume of batch } (V_B) = \frac{(A)}{(B)} \cdot (C) + \frac{W_{CA}}{62.3\, g_s} \ldots\ldots \quad \text{(E)}$$

The weight of coarse aggregate in a representative sample of concrete is then the ratio of the volume of the measure to that of the batch, multiplied by the weight of coarse aggregate in the batch. This is the value of W_s in Eq. 3, and is

$$W_s = \frac{V_M}{V_B} \cdot W_{CA} \ldots\ldots \quad (4)$$

The value of ΔW_s is therefore the weight of recovered coarse aggregate less the weight of the coarse aggregate in a representative sample, or

$$\Delta W_s = (W_s + \Delta W_s) - W_s \ldots\ldots \quad (5)$$

ΔW_s is the sampling error as previously defined, and is generally more significantly expressed as a percentage of W_s than by its value in pounds.

To determine the error in the air content as derived from a nonrepresentative sample of concrete, let W_B be the weight of the batch, and let V_{TB} be the theoretical volume of the batch without air. Then by the volumetric formula for air

Computed volume of air in sample

$$= V_M - \frac{W_M}{W_B} \cdot V_{TB} \ldots \quad \text{(F)}$$

Substituting the value of W_M from Eq. 2

Computed volume of air in sample

$$= V_M - \frac{W_T}{W_B} \cdot V_{TB} - \frac{K \Delta W_s}{W_B} \cdot V_{TB} \ldots \quad (6)$$

The first two terms on the right hand side of Eq. 6 give the air that would be present in the sample if it were strictly representative of the batch, and the third term is therefore the amount by which the air in the nonrepresentative sample is in error. This error, like that in the sample weight, is seen to be directly proportional to ΔW_s. The per cent error in the air is seen to be

$$p = 100 \frac{K \Delta W_s}{W_B \cdot V_M} \cdot V_{TB} \ldots\ldots \quad (7)$$

It is perhaps to be emphasized here that in speaking of errors in air determinations we are expressing the errors as "percentage points," additive or subtractive to the percentages as computed.

TABLE I.—AIR DETERMINATIONS CORRECTED FOR SAMPLING ERRORS.

Batch	0.1-cu. ft. Measure			0.5-cu. ft. Measure			Percentage Points Difference in Air
	Air, per cent computed	Sampling Error, per cent	Air, per cent corrected	Air, per cent computed	Sampling Error, per cent	Air, per cent corrected	
	(a)	(b)	(c)	(d)	(e)	(f)	(f)−(c)
P1 A1	2.02	0.44	2.07	1.39	6.49	2.13	0.06
P1 A2	2.17	1.92	2.39	2.24	2.89	2.58	0.19
P1 A3	2.19	−0.91	2.08	2.41	−1.49	2.23	0.15
P3 B2	3.85	−3.26	3.43	3.73	−1.97	3.49	0.06
P3 B3	3.83	−3.69	3.37	3.51	0.39	3.56	0.19
P3 B4	3.91	−1.24	3.75	3.60	2.84	3.97	0.22
P9 A1	2.85	−3.76	2.41	2.48	1.00	2.59	0.18
P9 A2	3.11	−4.21	2.62	2.46	3.46	2.87	0.25
P9 A3	3.08	−0.25	3.06	3.12	0.25	3.15	0.09
PC 1	4.94	−0.73	4.85	4.84	1.78	5.09	0.24
PC 2	4.87	−0.62	4.77	4.74	2.62	5.11	0.34
PC 3	4.74	−2.54	4.41	4.11	2.39	4.44	0.03
Average		1.96			2.29		0.17

The computations involved in deriving the values for K, ΔW_s, V_{TB}, etc., are somewhat long, but they can be systematized for machine work in various ways. In general, the computations should be carried to four, and occasionally five, significant figures.

EXPERIMENTAL DETERMINATION OF SAMPLING ERRORS AND THEIR EFFECTS

Our first attempt at evaluation of sampling errors was made in the latter part of 1945 on a series of paving concretes containing 2-in. maximum crushed stone aggregate and having slumps of around 3 in. Concrete was mixed in an open pot-type tilting mixer of 3.5-cu. ft. capacity, and samples were taken directly from the batch without dumping. Unit weights were determined in the usual manner in 0.1- and 0.5-cu. ft. measures, the coarse aggregate in each sample being recovered and weighed, and the corrections to unit weight and air content being computed and applied as previously outlined. The twelve batches for which the data in Table I are given consisted of three similar mixes with each of four different cements. To save space, the data in Table I are confined to (1) air content as computed from observed sample weights, (2) per cent sampling error, (3) corrected air content, and (4) differences in corrected air content as determined in the two measures.

Perhaps the most striking feature of the foregoing data is the fact that both the maximum and average sampling errors were less in the 0.1 than in the 0.5-cu. ft. measure. Also, why were most of the errors negative in the former, and mostly positive in the latter? We have only conjectural explanation for these unexpected results, and one might be inclined to question the evidence except for the fact that the corrected air values are generally in excellent agreement. Thus in only one case (batch PC 2) is there a discrepancy in the corrected air determinations in the two measures as great as 0.3 per cent, as shown in the last column of the table. On the other hand, if one were to compare the corrected air values with the values computed in the usual manner, he would find 11 cases out of 24 in which the corrections were greater than 0.3 per cent, these corresponding to sampling errors which amounted to 2 per cent or more. The average error in air content is actually about 0.13 per cent for 1 per cent sampling error.

The last column of Table I shows that the average difference between the corrected air contents of the twelve batches as determined from the two measures is 0.17 percentage points—less air in the small samples. This seems to result from a difference in effective consolidation and suggests that the specified 25 roddings per layer in the 0.5-cu. ft. measure is inadequate for this type of concrete. As a matter of fact some liberties were taken, particularly in filling the 0.5-cu. ft. measure, to overcome what was felt to be the inadequacy of the specified rodding treatment. These consisted of increased amounts of rodding and of some jolting of the measure, but in spite of this, higher voids were found in the larger samples.

A more extensive series of tests on paving concretes was made in the fall of 1946 in connection with performance tests of a new 3.5 S Smith Tilting Mixer. In our limited experience this is an excellent mixer, but it has some drawbacks for laboratory work. The chief of these is a large blade surface which excessively robs the mix of mortar, and for this reason the four small deflecting blades were removed, leaving the four main mixing blades. With care to recover all the mortar that could readily be recovered, some 5 per cent of the mortar in $2\frac{1}{2}$-cu. ft. batches of pavement-type concrete was left in the mixer, when it was clean at the start. This loss of mortar should tend to give a higher ratio of coarse aggregate to mortar than actually existed in the test batches, and the conditions were therefore such as to provide a critical test of the evaluation of sampling errors, and of the correction of resulting errors in unit weight and air content.

The data in Table II include all batches that were made in testing the general performance of the mixer except a few that were preliminary trials. Batches were for the most part of $2\frac{1}{2}$-cu. ft. size containing $1\frac{1}{2}$-in. maximum crushed stone aggregate and approximately 6 bags per yard, at 3-in. slump. A few of the batches contained 2-in. maximum aggregates, and some were primed from previous batches, although not effectively in all cases. There were also changes in fine aggregates, air entraining admixtures, etc. The main feature of the program, so far as the magnitude of sampling errors and of the resulting corrections was concerned, was the use of 0.1, 0.2, and 0.5-cu. ft. measures for comparative tests. As previously explained, sampling errors were determined by recovering and weighing the stone from the test samples. The 0.2-cu. ft. measure used was the base section of a Klein-Walker air meter, and volumetric results therefrom were compared with the gravimetric values resulting from tests made on the same samples.

One of the most interesting results from this series of tests is the diversity and distribution of the sampling errors obtained in the three sizes of measures—shown in columns (*a*), (*b*), and (*c*) of Table II. It is seen that sampling errors up to about 7 per cent occur in all three measures, and that the grand average values of the sampling errors is about 3.3 per cent in each of the three. This confirms the indications from the first series of tests and seems to be contrary to the general assumption that sampling errors are kept at a minimum by using 0.5 cu. ft. or larger measures for concretes containing $1\frac{1}{2}$-in. maximum aggregate or larger. In fact the results suggest that the general rule for the size of test cylin-

TABLE II.—DATA ON THE EFFECTS OF SAMP-

Batch	Maximum Size of Aggregate, in.	Slump, in.	Mixer Clean or Primed	Sampling Error, per cent			Corrected Unit Weight of Concrete, lb. per cu. ft.			
				Size of Measure, cu. ft.			Size of Measure, cu. ft.			
				0.5	0.2	0.1	0.5	0.2	0.1	Average
				(*a*)	(*b*)	(*c*)	(*d*	(*e*)	(*f*)	(*g*)
No. 5	1.5	2.5	C	3.41	6.75	9.36	152.15	152.08	152.64	152.29
No. 6		3.8	P	0.20	−4.15	−2.99	152.00	151.76	151.92	151.89
No. 7		2.4	C	1.42	5.86	4.13	151.76	152.15	152.28	152.06
No. 10		1.9	C	1.81	2.23	0.97	150.37	150.23	151.44[a]	150.68
No. 11		2.0	P	−1.42	−1.18	−5.38	150.09	150.23	150.33	150.22
No. 12		2.0	C	3.86	3.92	2.89	146.97	146.73	147.32	147.01
No. 13		2.6	C	6.02	2.96	1.80	144.69	143.89[a]	144.96	144.51
No. 14		2.9	P	3.34	3.30	0.12	145.60	145.05	145.25	145.30
No. 15		3.8	C	7.64	1.29	7.06	143.94	143.37	144.01	143.77
No. 16		1.8	P	2.73	−2.18	−0.32	147.01	146.94	147.48	147.14
No. 17		3.6	C	4.25	5.41	−1.88	147.49	147.23	147.34	147.35
No. 18		2.5	C	4.04	1.92	4.60	147.69	147.63	147.99	147.77
No. 19		3.1	C	2.15	4.87	2.28	147.21	146.87	147.14	147.07
No. 20	2	3.5	C	3.89	0.84	4.96	149.48	149.33	149.30	149.37
No. 21		2.6	C	4.14	2.86	1.81	150.67	150.98	150.94	150.86
No. 22		3.1	P	−1.48	−1.62	−1.13	150.30	150.88	150.84	150.67
No. 23		2.6	C	3.53	7.51	4.24	150.78	151.35	150.88	151.00
No. 24		3.5	P	3.15	−2.95	−4.24	150.14	150.37	150.50	150.34
	Average Values									
	For 13 1.5-in. maximum aggregate mixes			3.25	3.54	3.37				
	For 5 2-in. maximum aggregate mixes			3.24	3.16	3.28				
	For 12 C mixes			3.84	3.87	3.83				
	For 6 P mixes			2.05	2.56	2.36				
	Grand average values			3.25	3.43	3.34	148.96	148.93	149.14	

[a] These values are quite out of line with the corresponding values obtained in the other two measures and suggest that 13 are omitted from the grand averages of columns (*d*), (*e*), (*f*), and (*k*), (*l*), (*m*).

ders may be applied for test samples, namely, that the diameter of the measure should be three or four times the maximum size of the aggregate. Nevertheless one may argue that both common sense and the laws of chance indicate that sampling errors should diminish with increasing size of sample. The answer to this may be that the operator tries to fill a measure with trowelsful or scoopsful or shovelsful of concrete that are in themselves judged to be as nearly representative of the batch as possible. If there is a tendency to introduce a systematic error in loading the trowel, scoop or shovel, this will be more or less in evidence, regardless of the size of measure. Whatever the true explanation may be, there seems, in our laboratory, at least, little advantage in using the large size measures, and none at all if corrections are to be made for the sampling errors.

LING ERRORS IN PAVEMENT-TYPE CONCRETE.

Error in Unit Weight, per cent			Corrected Air (Gravimetric) in Concrete, per cent				Air by Klein-Walker Meter		Percentage Points Error in Air, Gravimetric			Percentage Points Difference (*p*) − (*l*)
Size of Measure, cu. ft.			Size of Measure, cu. ft.				Percentage Points Error	Corrected per cent Air	Size of Measure, cu. ft.			
0.5	0.2	0.1	0.5	0.2	0.1	Average			0.5	0.2	0.1	
(*h*)	(*i*)	(*j*)	(*k*)	(*l*)	(*m*)	(*n*)	(*o*)	(*p*)	(*q*)	(*r*)	(*s*)	(*t*)
0.43	0.85	1.15	2.33	2.38	2.02	2.24	−0.13	2.16	−0.42	−0.82	−1.12	−0.22
−0.01	−0.51	−0.36	2.03	2.18	2.07	2.09	0.08	2.32	0.02	0.50	0.36	0.14
0.18	0.74	0.52	2.05	1.80	1.72	1.86	−0.11	2.07	−0.18	−0.73	−0.51	0.27
0.23	0.29	0.12	3.38	3.47	2.69[a]	3.18	−0.08	3.95	−0.22	−0.27	−0.11	0.48
−0.18	−0.15	−0.68	3.27	3.18	3.11	3.19	0.04	3.83	0.18	0.14	0.66	0.65
0.56	0.57	0.41	5.12	5.27	4.90	5.10	−0.18	5.48	−0.52	−0.53	−0.39	0.21
0.93	0.47	0.27	6.23	6.75[a]	6.06	6.35	−0.16	6.78	−0.86	−0.43	−0.26	0.03
0.49	0.50	0.02	5.46	5.81	5.68	5.65	−0.17	6.26	−0.47	−0.47	−0.02	0.45
1.20	0.21	1.11	7.16	7.52	7.11	7.26	−0.08	7.63	−1.12	−0.19	−1.03	0.11
0.39	−0.31	−0.07	4.40	4.44	4.09	4.31	0.09	4.83	−0.37	0.30	0.04	0.39
0.60	0.77	−0.26	4.86	5.03	4.96	4.95	−0.24	5.42	−0.57	−0.73	0.25	0.39
0.58	0.27	0.64	4.84	4.88	4.65	4.79	−0.08	5.19	−0.54	−0.26	−0.61	0.31
0.31	0.69	0.32	5.15	5.37	5.20	5.24	−0.22	5.56	−0.29	−0.66	−0.31	0.19
0.56	0.12	0.72	3.93	4.02	4.04	4.00	−0.04	4.43	−0.54	−0.12	−0.69	0.41
0.56	0.39	0.25	3.30	3.10	3.12	3.17	−0.10	3.52	−0.56	−0.38	−0.24	0.42
−0.21	−0.23	−0.16	3.44	3.07	3.09	3.20	0.06	3.64	0.20	0.22	0.15	0.57
0.49	1.01	0.58	3.16	2.79	3.10	3.02	−0.24	3.33	−0.47	−0.99	−0.57	0.54
0.45	−0.40	−0.58	3.57	3.42	3.34	3.44	0.11	3.85	−0.43	0.40	0.57	0.43
0.47	0.49	0.46							0.44	0.46	0.44	
0.45	0.43	0.46							0.44	0.42	0.44	
0.55	0.53	0.53							0.52	0.51	0.51	
0.29	0.35	0.31							0.28	0.34	0.30	
0.46	0.47	0.46	4.00	4.02	3.89		0.12		0.44	0.45	0.44	0.32

ome observational error was responsible for them. On this account all the unit weight and air values for batches 10 and

Columns (*h*), (*i*), and (*j*) show the percentage errors in unit weight resulting from the sampling errors. The grand average values in the last line are practically identical for the three sizes of measures, and show that under the conditions of these tests each per cent sampling error produced an error in unit weight of about 0.14 per cent.

It may not be readily apparent that the errors in the air determinations, in percentage points, due to sampling errors, are about identical with the percentage errors in unit weight, but such is the case. Percentage error in unit weight as derived from Eq. 2 is $\frac{100}{W_T}\cdot K\Delta W_s$, and that for air, in percentage points, is $-\frac{100}{W_B V_M/V_{TB}}\cdot K\Delta W_s$ from Eq. 7. Now $W_T = W_B \cdot V_M/V_B$, and therefore the coefficients of $K\Delta W_s$ in the two expressions are very nearly equal, differing only in sign and by the difference in values of V_B and V_{TB}. It follows that the error in air is smaller than the error in unit weight by an amount equal to the latter multiplied by the decimal air content of the concrete, and where these errors are small, the difference may be considered negligible. The computation for one therefore serves practically for the other, which is shown by the close agreement of corresponding values in columns (*h*), (*i*), and (*j*) with those in columns (*q*), (*r*), and (*s*).

It may be noted in column (*o*) that the errors in the determinations of air by the Klein-Walker meter, due to sampling errors, are much smaller than those due to the same cause in the gravimetric air determinations. Thus the average per cent error in the latter, using the 0.2-cu. ft. measure, was 0.45 per cent, (column (*r*)) while the average error in the Klein-Walker determinations, on the same samples of concrete, was only 0.12 per cent. This low susceptibility to the effects of sampling errors of the Klein-Walker method (or any other method of direct volume measurement) as compared to the comparatively high susceptibility of the gravimetric method, is a point very much in its favor.

The general relations between sampling errors and errors in unit weight and air determinations are shown in Fig. 1.

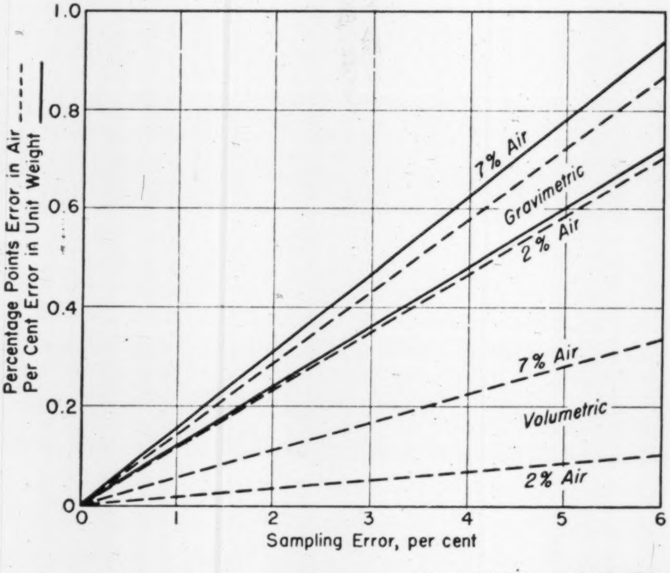

FIG. 1.—Effect of Sampling Error on Uncorrected Unit Weight and Air Content.

Based on the series of tests summarized in Table II, the effect of sampling errors is shown for concretes containing 2 and 7 per cent air, both for gravimetric and volumetric determinations. In the type of concrete used in these studies, the diagram shows that for 7 per cent air the gravimetric errors are about 2.5 times as large as the volumetric errors, while for 2 per cent air they are nearly 7 times as large. In the normal range of 3 to 6 per cent air, it is seen that an average sampling error of 2 per cent would produce an error in the volumetric air determination of less than 0.1 percentage points.

But regardless of these advantages of the volumetric method, our Klein-Walker meter gave a higher average air content than the gravimetric determinations on the same samples by 0.32 percentage points. The difference between the Klein-Walker values in column (*p*) and the gravimetric values in column (*l*) are shown in column (*t*), and it is seen that in only one case was there a negative difference. This discrepancy in the Klein-Walker determinations and the gravimetric determinations is considerable, and suggests that small shrinkage or specific gravity errors in the concrete mixtures or small calibration errors in the apparatus were still present, even though special efforts were made to eliminate them.

The effect of priming the mixer in 6 of the 18 batches reported in Table II was to reduce the size of the sampling errors, as shown by the average values near the bottom of columns (*a*), (*b*) and (*c*). This was to be expected since the batches from the unprimed mixer were deficient in mortar. This emphasizes the necessity for priming, or for some other scheme having an equally beneficial effect, in order to minimize sampling errors and their effects. Our experience in this study suggests that if the loss of mortar in any given mixing operation is known or can be estimated approximately, an economical procedure would be to design a mix in the usual way, and then deliberately add about the amount of extra mortar that would be retained in the mixer. This would eliminate the priming operation and reduce the sampling error to the same order of magnitude that it would have if the undoctored batch could be entirely recovered from the mixer.

The 2-in. maximum aggregate in five batches as compared with the $1\frac{1}{2}$-in. maximum aggregate in thirteen batches had no discernible effect on the magnitude of the sampling errors in the various size measures. As shown near the bottom of columns (*a*), (*b*), and (*c*); (*h*), (*i*), and (*j*); and (*q*), (*r*), and (*s*) neither the average sampling errors nor the resulting average errors in unit weights and air contents were larger in the samples containing the coarser aggregate.

The generally close agreement of the corrected unit weights and air determinations, shown in columns (*d*), (*e*), and (*f*) and in (*k*), (*l*), and (*m*) of Table II, demonstrates that the corrections for sampling errors can be expected to give consistent results regardless of size of measure used for the concrete test sample. This fact suggests that more accurate results at no greater expenditure of time and labor may be secured in laboratory studies of air entrainment by the use of a relatively small measure, by determining the sampling error and correcting for it, than in trying to minimize errors by priming the mixer, by using larger measures, and disregarding the sampling error. However, when the recovery of the coarse aggregate from test samples is impracticable, the mixer should be primed with a wasted batch, or the mortar should be increased by an amount which is expected to be retained in the mixer. The sampling error will not be entirely eliminated by either of these procedures, but it should be smaller than it would be if no attempt is made to offset the loss of mortar.

The average values of the corrected unit weights and air contents of 16 of the test batches, shown in the last line of columns (*d*), (*e*), and (*f*) and (*k*), (*l*), and (*m*), indicate that slightly lower weights and slightly higher air contents were given by the 0.5-cu. ft. measure than by the 0.1-cu. ft. measure. The difference is small in this series (Table II), but is evidence of less effective elimination of voids in the large measure, which was somewhat more marked in the first series (Table I).

SUMMARY AND CONCLUSIONS

This study of sampling errors (defined as the excess or deficiency of coarse aggregate in nonrepresentative samples taken from test batches of concrete) shows that such errors are practically always present in some degree and are frequently the source of considerable errors in unit weight and air determinations. An algebraic analysis shows how sampling errors may be evaluated, and what relation they bear to the concomitant errors in unit weight and air content.

Test results from 30 batches of pavement-type concrete confirm the theoretical analysis and demonstrate that dependable and sufficiently accurate values for unit weight or entrained air may be obtained regardless of the size of test sample (if 0.1 cu. ft. or larger), or regardless of the magnitude of any sampling error that is likely to occur.

A number of practical laboratory procedures in the testing of air-entrained concrete have suggested themselves in the course of this study:

1. It has been proposed that the determination of sampling errors in laboratory studies will yield more accurate test results, with little or no sacrifice of time or labor, than the present prescribed methods of testing and sampling concrete batches.
2. It has been suggested that the diameter and height of a suitable container for test samples of concrete need not be more than three or four times the maximum size of aggregate.
3. Both the theoretical analysis and the test results show that the percentage error in unit weights, and the percentage points error in air, due to the sampling error, are numerically the same except for minute and negligible differences. This materially reduces the computation involved in deriving the corrections resulting from sampling errors.
4. Whenever it is impracticable to determine sampling errors, priming of the mixer or some other equally effective means of compensating for mortar loss in the mixer should not be omitted in the test procedure.
5. It has been shown that direct measurements of entrained air, such as the Klein-Walker method, are much less susceptible to the effects of sampling errors than the commonly used gravimetric method.
6. In this investigation, the Klein-Walker meter gave slightly higher values for the entrained air than were obtained on identical samples of concrete by the gravimetric method.

DISCUSSION

Mr. Delmar L. Bloem[1] (*presented in written form*).—For some time past the Research Laboratory of the National Ready Mixed Concrete Association at the University of Maryland has been cognizant of the rather large sampling errors resulting from the mortar being retained in the mixer when a batch of concrete is discharged. Since the mixer used in this laboratory is very small (1 cu. ft. capacity tilting type) and hence has a large interior surface area with respect to the volume of concrete mixed, a rather large proportion of the mortar is retained in the mixer. Tests have shown that in small batches as much as 10 per cent of the mortar might be held back—enough to produce an error in air content determination of 0.6 or 0.7 of a percentage point. Similarly, computed values of cement content, water content and ratio of fine to coarse aggregate were found to be in error. For 10 per cent holdback of an average mix the computed cement factor was found to be 0.4 sack per cu. yd. too high and the computed water content 2.3 gal per cu. yd. too high. The sand content expressed as a percentage of the total aggregate was computed 3.5 percentage points too high. Although errors such as these due to mortar holdback would certainly not be encountered in the field, they would be very likely to occur in the laboratory and would be very significant where control within close tolerances is necessary.

In order to minimize this sampling error, a method of "buttering" the mixer has been adopted as standard procedure by our laboratory. It consists simply of providing each batch of concrete with sufficient extra mortar to compensate for what is retained in the mixer. The steps in the method are essentially as follows:

1. The test batch of concrete is designed in accordance with accepted design procedures and the design checked by means of trial batches.
2. The quantities of mortar ingredients (sand, cement, water and admixture when used) are increased by a certain percentage depending upon the size of batch. For example, in a 0.5-cu. ft. batch, these quantities are increased by 8 per cent since experience has shown that approximately this percentage of the mortar is retained in the mixer.
3. After being mixed the concrete is discharged from the mixer (care being exercised that all coarse aggregate is removed) and the entire batch is weighed.
4. The weight of the batch is adjusted to the original design weight either by removing mortar from the batch or adding mortar which has been scraped from the mixer.

The question might be raised as to whether the "butter" left in the mixer is of the same composition as the whole of the mortar in the batch. Although this problem has been investigated only sketchily, indications are that the differences in composition between the "butter" and the mortar in the batch are so slight as to have no discernible effect on the composition of the concrete.

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[1] Associate Research Engineer, National Ready Mixed Concrete Association, Washington, D. C.

It is recognized that this is only one of several possible methods of coping with the problem of sampling errors and that other laboratories may find means which are more satisfactory for their individual type of operation. The method just described does not eliminate the errors caused by failure to secure a representative sample from the concrete for unit weight or air content determination. However, it is believed that experience and care in sampling can go far toward minimizing errors from this source. In the laboratory, at least, these same errors would also be present in securing samples for test specimens; therefore, for most work it would seem inconsistent to make meticulous corrections in the computed characteristics of the concrete when it is impossible to know whether or not the concrete in test specimens has these same characteristics as accurately as they were computed. Another possible objection to the method described by Pearson and Helms is the fact that, in carefully controlled laboratory research, it may not always be desirable, even if feasible, to remove a portion from each batch of concrete for the determination of sampling error. The advantage gained by a slightly more accurate determination of air content might be more than offset by alterations in the properties of the remaining concrete caused by removal of an unrepresentative sample. This would be especially true for the small sized batches employed in our own laboratory.

MR. E. M. BRICKETT.[2]—I should like to ask the authors, and Mr. Bloem too, just what method was used in striking off the concrete in making the unit weight determination.

Some operators, in screeding off the unit container, tend to push coarse aggregate down into the container and then remove mortar in the screeding process. Of course, such practice results in a change in the composition of the concrete.

Recently I watched some tests in which the operator used a heavy glass plate to strike off the measure, bearing down heavily on the plate, rotating the plate and moving it back and forth to remove the excess concrete in the measure. There were at least two objectionable points to this procedure: one, of course, was that it was only the finest portion of the mortar which was removed, thus changing the composition of the concrete in the measure. It was also very noticeable that when air-entraining concrete was screeded in this manner the pressure exerted on the glass plate evidently compressed the concrete slightly. As the operator took pressure off the glass plate, it rose, possibly $\frac{1}{32}$ in. Thus the actual quantity of air-entrained concrete in the measure exceeded the volume of the measure.

MR. J. C. PEARSON (*author's closure by letter*).—The discussion by Mr. Bloem is appreciated as an example of good practice in the technique of mixing and sampling of concrete test batches in the laboratory. As he and others have shown, the mortar retained in the mixer cannot be disregarded, and the deliberate increase in the mortar content of the designed batch is, in the authors' opinion, probably the most feasible way of compensating for the loss in the mixer. However, with this source of error properly taken care of, sampling errors may still be present that are likely to be larger than many technicians would suspect, even with much "experience and care in sampling."

The authors would like to suggest that other workers in this field could study the sampling error problem to advantage by making a considerable number of repeat tests on unit weights of samples of concrete taken from 1-cu. ft. or larger machine mixed batches of pavement-type concrete, and by recovering and weighing the coarse aggregate from the samples. Such tests would give data (1) for an estimate of maximum and average value of sampling error, and (2) for the degree of correlation between sampling errors and variations in unit weight.

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[2] Cement Division, Dewey and Almy Chemical Co., Cambridge, Mass.

The paper was not intended to emphasize unduly the importance of these sampling errors, except in their effect on air determinations by the gravimetric method. The effect of these errors, when proper precautions have been taken to minimize them, is not large on volumetric air determinations, nor on the consistency and strength of the concrete. In fact if one's faith in the water-cement ratio–strength relation is steadfast, a little more or a little less mortar in the concrete test specimens should have no effect on strength at all. But Mr. Bloem's caution against straining at gnats and swallowing camels is well taken, for there are certainly other errors in the entire process of testing concrete that are quite as significant as the sampling errors discussed in the paper.

In answer to Mr. Brickett's question, any carelessness in filling and screeding the measure in the manner he describes is another source of error affecting the mortar-coarse aggregate ratio. We prefer the glass plate method of screeding, but care is always taken to fill the measure in such manner that no large excess or deficiency of concrete occurs when the concrete has been rodded. If such occurs, the endeavor is to add or remove *concrete* (not mortar alone) with the end of a plasterer's trowel to bring it closely to the level of the top of the measure before screeding with the glass plate.

THE EFFECT OF BLENDS OF NATURAL AND PORTLAND CEMENT ON PROPERTIES OF CONCRETE*

By A. G. Timms,[1] W. E. Grieb,[1] and George Werner[1]

Synopsis

The paper summarizes the results of tests of concrete made with seven natural cements and a slag cement blended with each of three different nonair-entraining portland cements. Comparable tests of concrete with two air-entraining portland cements were also made. Some of the natural cements contained air-entraining material in sufficient amounts to produce air-entraining concrete when blended with nonair-entraining portland cements. Others entrained very little air.

It is concluded that the increased resistance to freezing and thawing of concretes containing blends of portland cement and certain natural cements, in the proportions used in this investigation, is due to the fact that the concrete produced with these blends contained entrained air.

An investigation of the use of blends of natural and portland cement for improving the quality of concrete was reported in 1938 by the Public Roads Administration.[2] That investigation, prompted by the increasing use of blends in the field, was limited to the use of one natural cement in the case of the laboratory fabricated specimens and to two natural cements in the case of cores drilled from pavements and tested in the laboratory.

The present report summarizes the results of a considerably expanded laboratory investigation begun in January 1942 and involved the use of seven natural cements and one slag cement, each of which was blended with each of three plain or nonair-entraining portland cements.[3] In addition, comparable tests were made with the three plain portland cements as well as with the air-entraining counterparts of two of the them. Some of the natural cements contained sufficient air-entraining material to produce air-entraining concrete, while others produced little or no additional air.

General Test Procedures

In construction, the general procedure has been to use a blend consisting of one bag of natural cement with either five or six bags of portland cement. In these tests all blends were in the proportion of 14 per cent natural cement to 86 per cent portland cement by weight. This is about the proportion of one bag of natural cement weighing 80 lb. to five bags of portland cement weighing 94 lb. each.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Senior Materials Engineer, Associate Highway Engineer, and Associate Materials Engineer, respectively, Division of Physical Research, Public Roads Administration, Washington, D. C.

[2] W. F. Kellermann and D. G. Runner, "The Effect of Using a Blend of Portland and Natural Cement on Physical Properties of Mortar and Concrete," *Proceedings*, **Am. Soc. Testing Mats., Vol. 38, Part II, p. 329 (1938); also *Public Roads*, October, 1938.**

[3] Certain of the freezing-and-thawing data from Series 2 of the present report were presented by W. F. Kellermann, "Effect of Blended Cements and Vinsol Resin-Treated Cements on Durability of Concrete," ***Journal*, Am. Concrete Inst., June, 1946.**

TABLE I.—CHEMICAL AND PHYSICAL PROPERTIES OF PORTLAND CEMENTS.

	Cement A1	Cement A2	Cement B1	Cement B2	Cement C1
Chemical Analyses, per cent					
Silica (SiO_2)	21.60	21.60	23.90	23.90	21.55
Alumina (Al_2O_3)	6.14	6.29	4.27	4.14	6.02
Ferric Oxide (Fe_2O_3)	2.56	2.56	3.28	3.36	3.68
Lime (CaO)	63.90	63.85	64.60	64.65	63.90
Magnesia (MgO)	2.93	2.93	1.09	1.10	1.10
Sulfuric anhydride (SO_3)	1.58	1.61	1.41	1.42	1.58
Sodium and potassium oxide ($Na_2O + K_2O$)	0.68	0.54	0.64	0.61	0.68
Loss on ignition	0.62	0.64	0.84	0.78	1.42
Chloroform soluble material[a]	0.007	0.035	0.006	0.040	0.010
Computed Compound Compositions, per cent					
Tricalcium silicate (C_3S)	46	45	44	45	46
Dicalcium silicate (C_2S)	27	28	36	35	27
Tricalcium aluminate (C_3A)	12	12	6	5	10
Tetracalcium alumino-ferrite (C_4AF)	8	8	10	10	11
Calcium sulfate ($CaSO_4$)	2.7	2.7	2.4	2.4	2.7
Physical Properties					
Apparent specific gravity	3.17	3.21	3.17	3.22	3.19
Specific surface (Wagner), sq. cm. per g.	1730	1670	1690	1735	1910
Autoclave expansion, per cent	0.15	0.17	0.01	0.02	0.05
Normal consistency, per cent	23.5	23.0	24.5	24.0	25.0
Time of setting (Gillmore test)					
Initial set, hr., min.	3—10	2—40	3—45	3—30	3—30
Final set, hr., min.	4—45	4—40	5—00	5—25	5—00
Tensile strength (1:3 mortar), psi.					
3 days	305	280	285	225	295
7 days	415	370	325	305	360
28 days	480	490	465	385	445
Compressive strength (1:3 mortar), psi.					
3 days	2015	1805	1695	1420	2050
7 days	3645	3495	2870	2395	3515
28 days	5450	5020	4715	4045	5615

[a] A.S.T.M. Method C 114-42 used.

TABLE II.—CHEMICAL AND PHYSICAL PROPERTIES OF NATURAL AND SLAG CEMENTS.

	Cement I	Cement IIa	Cement IIb	Cement III	Cement IVa	Cement IVb	Cement V	Cement VI
Chemical Analyses, per cent								
Silica (SiO_2)	24.15	23.60	23.55	18.15	25.25	25.25	23.75	30.50
Alumina (Al_2O_3)	5.28	7.19	7.40	4.57	5.50	5.50	5.30	9.41
Ferric oxide (Fe_2O_3)	4.32	2.56	2.40	2.08	2.40	2.40	2.80	1.44
Lime (CaO)	33.45	33.05	33.00	46.80	34.45	34.30	34.45	51.15
Magnesia (MgO)	21.65	20.77	20.68	10.45	21.62	21.96	21.65	1.83
Sulfuric anhydride (SO_3)	1.30	1.61	1.96	1.78	2.35	2.25	2.30	0.45
Sodium and potassium oxide ($Na_2O + K_2O$)	1.78	1.78	1.83	1.06	3.00	2.93	3.41	...
Loss on ignition	7.90	9.54	9.29	15.07	5.50	5.35	6.40	3.85
Insoluble residue	14.25	13.85	13.85	6.30	11.15	11.05	11.65	...
Chloroform soluble material[a]	0.05	0.04	0.32	0.33+[b]	0.08	0.13	0.18	...
Physical Properties								
Apparent specific gravity	3.06	2.90	2.88	2.86	3.03	3.05	3.00	2.80
Specific surface, (Wagner), sq. cm. per g.	2310	2350	2445	3030	2285	2035	2565	2770
Autoclave expansion, per cent	3.8[c]	1.8	2.5	4.2	15.0	15.0	5.5[c]	−0.1
Normal consistency, per cent	29.0	31.0	32.0	34.0	36.0	38.0	32.0	32.0
Time of setting (Gillmore test)								
Initial set, hr., min.	0—45	3—15	1—10	2—15	1—00	0—55	1—10	2—35
Final set, hr., min.	1—15	5—30	2—35	5—15	1—45	1—45	1—50	5—00
Tensile strength (1:2 mortar), psi.								
3 days	40	35	65	80	40[d]	65[d]	75	60[d]
7 days	50	65	115	125	90[d]	90[d]	125	215[d]
28 days	70	170	235	270	210[d]	200[d]	195	380[d]
Compressive strength (1:2 mortar), psi.								
3 days	135	175	360	510	185[d]	215[d]	540	275[d]
7 days	150	365	655	765	305[d]	285[d]	700	1060[d]
28 days	430	1160	1605	1790	855[d]	710[d]	1150	2490[d]

[a] A.S.T.M. Method C 114-42 used.

[b] All chloroform soluble material not extracted.

[c] Specimen warped.

[d] 1:3 mortar used.

The three portland cements used in the investigation were designated as cements A, B, and C. All of them were nonair-entraining and as such were designated as cements A1, B1 and C1. Flake Vinsol resin was interground with both cements A and B and produced air-entraining portland cements designated as A2 and B2. There was no air-entraining counterpart for cement C. The natural cements used in the investigation were designated as cements I to V, inclusive, and the slag cement as cement VI. Two lots of each of natural cements II and IV, differing considerably in the amount of chloroform-soluble material in the cement, were tested.

The cements containing the smaller amounts of chloroform-soluble material were designated as IIa and IVa, while the cements containing the larger amounts were designated as IIb and IVb. The results of the chemical and physical tests on the cements are given in Tables I and II.

TABLE III.—MIX DATA.[a]

Cement[b]	Series 1						Series 2		Series 3	
	Water Content,[c] gal. per bag	W_c[d]	Slump, in.	Weight of Fresh Concrete, lb. per cu. ft.	Actual Cement Content,[c] bags per cu. yd.	Calculated Air Content, per cent	Water Content, gal. per bag	Slump,[c] in.	Water Content, gal. per bag	Slump,[c] in.
A1	5.2	0.152	2.6	149.6	6.0	1.3	6.1	3.3	4.8	3.3
A2	5.0	0.146	2.8	148.1	5.9	2.8	5.7	3.1	4.6	3.7
A1 + I	5.2	0.150	2.9	148.0	5.9	2.3	6.1	3.8	4.8	3.1
A1 + IIa	5.2	0.152	2.6	149.2	5.9	1.5	6.1	3.4	4.8	2.8
A1 + IIb	5.1	0.144	3.1	143.6	5.7	5.3	5.8	3.9	4.7	3.0
A1 + III	5.2	0.149	3.0	146.8	5.8	3.0	6.0	3.8	4.8	2.6
A1 + IVa	5.2	0.151	2.8	146.8	5.8	3.0	...	...	4.9	2.7
A1 + IVb	5.2	0.146	2.7	143.8	5.7	5.1	...	...	4.8	2.8
A1 + V	5.2	0.149	2.9	146.5	5.8	3.3	6.0	4.0	4.8	3.3
A1 + VI	5.2	0.152	3.0	149.3	6.0	1.3	6.0	3.2	4.8	3.5
B1	5.1	0.150	2.8	150.1	6.0	1.2	5.9	3.5	4.8	2.8
B2	4.9	0.141	3.0	148.0	5.9	3.1	5.6	3.8	4.6	3.3
B1 + I	5.1	0.149	2.8	148.6	5.9	2.1	6.0	3.3	4.8	3.1
B1 + IIa	5.1	0.150	2.4	149.7	6.0	1.3	6.0	3.1	4.8	3.1
B1 + IIb	5.0	0.143	2.8	144.8	5.8	4.6	5.7	3.1	4.7	2.5
B1 + III	5.1	0.147	2.7	147.4	5.9	2.8	5.8	3.3	4.8	2.5
B1 + IVa	5.2	0.150	2.8	147.3	5.9	2.8	...	...	4.9	3.1
B1 + IVb	5.2	0.147	2.8	144.5	5.8	4.6	...	...	4.9	2.9
B1 + V	5.1	0.147	2.7	147.0	5.9	3.1	5.9	3.6	4.8	2.8
B1 + VI	5.1	0.150	2.9	149.5	6.0	1.4	5.9	3.0	4.8	3.3
C1	5.2	0.152	3.0	149.4	6.0	1.5	...	...	...	...
C1 + I	5.2	0.150	2.9	148.1	5.9	2.3	...	...	...	...
C1 + IIa	5.2	0.151	2.9	149.0	5.9	1.6	...	...	...	...
C1 + IIb	5.1	0.144	2.8	144.4	5.8	4.8	...	...	...	...
C1 + III	5.2	0.149	3.1	147.2	5.9	2.8	...	...	...	...
C1 + IVa	5.2	0.151	3.1	146.8	5.8	3.1	...	...	...	...
C1 + IVb	5.2	0.149	2.8	144.2	5.7	4.8	...	...	...	...
C1 + V	5.2	0.149	2.9	146.7	5.8	3.2	...	...	...	...
C1 + VI	5.2	0.151	2.7	148.9	5.9	1.7	...	...	...	...

[a] Mix by dry weight, lb., for series 1,—94:166:369
for series 2,—94:194:339
for series 3,—94:177:358.

[b] Maximum size coarse aggregate series 1 and 3, 1½-in., series 2, 1-in.
Where natural or slag cement was used, proportions were as follows: 86 per cent portland, 14 per cent natural or slag cement by weight.

[c] Where natural or slag cement was used, 94 lb. considered as one bag.

[d] Water per unit volume of concrete based on actual yield tests.

All mixes were designed on the basis of using a nonair-entraining portland cement and a constant consistency as measured by the slump test. No adjustments were made in the proportions when using air-entraining portland cements or the blending cements. As a result, the proportions of cement to aggregate by weight were the same for all cement combinations. Observations were made of the workability of all concrete mixes. Workability varied considerably. All of the air-entraining mixtures produced more workable concrete than those which contained only small quantities of air.

The amount of water per unit volume of concrete was calculated from actual yield tests. Differences in water contents reflect differences of the volume of air in the different mixes as well as slightly different water requirements of the various cements.

Series 1. Strength Tests:

Potomac River sand and gravel, $1\frac{1}{2}$-in. maximum size, were used as aggregates in a typical paving mix containing six sacks of cement per cubic yard. The fineness modulus of the sand was 2.54. Mix data are given in Table III. Test specimens were 6 by 12-in. cylinders for compression and 6 by 6 by 20-in. beams for flexure. The specimens were moist cured and tested at ages ranging from 3 days to 4 yr.

Series 2. Drying Shrinkage and Freezing-and-Thawing Tests:

Potomac River sand and gravel were used as aggregates. The maximum size was limited to 1 in. because of the small size of the test specimens. The same sand was used as that in series 1. Specimens were 3 by 4 by 16-in. beams. Two of the nonair-entraining portland cements, A1 and B1, the two air-entraining portland cements, and blends of the two nonair-entraining cements with five of the seven natural cements and the slag cement were included. Mix data are given in Table III. The nominal cement factor was 5.8 sacks per cubic yard. The specimens for drying shrinkage were stored in laboratory air maintained at a constant temperature of 70 ± 2 F., and constant relative humidity of 50 ± 2 per cent. Measurements were made at periodic intervals. Two groups of freezing-and-thawing tests were conducted. In the first group the tests were made on specimens cured continuously in moist air for 230 days before freezing was started. Sonic determinations were made at periodic intervals on these beams during freezing and thawing and flexural strength tests were made after 25 cycles of freezing and thawing. Companion beams, moist cured continuously, were also tested for flexural strength at the same age as those subjected to freezing and thawing.

In the second group of tests, specimens previously used for drying shrinkage measurements were, at the conclusion of the drying period, soaked in water for seven days, then frozen and thawed in water, using the same cycle as was used in the first group. Freezing and thawing was discontinued for 128 days at the end of 60 cycles, after which the cycles of freezing and thawing were again started.

Series 3. Effect of Segregation:

Experience has shown that the typical Potomac River sand grading used in Series 1 and 2 does not induce appreciable bleeding or water gain in concrete. On this account, a somewhat coarser sand was used in series 3 the fineness modulus being 3.01. The coarse aggregate was the same as in series 1. Two plain and two air-entraining portland cements and blends of all seven of the natural cements and the slag cement with the two plain cements were used. Mix data are given in Table III. Specimens were slabs 2 by 2 ft. and 8-in. in thickness. They were cast in forms 2 ft. long by 4 ft. wide and were screeded with an 8-in. screed weighing 100 lb. per ft. A tar paper separator installed full depth midway in the form made it possible to obtain two 2 by 2-ft. test slabs without restricting the lateral movement of the concrete caused by the

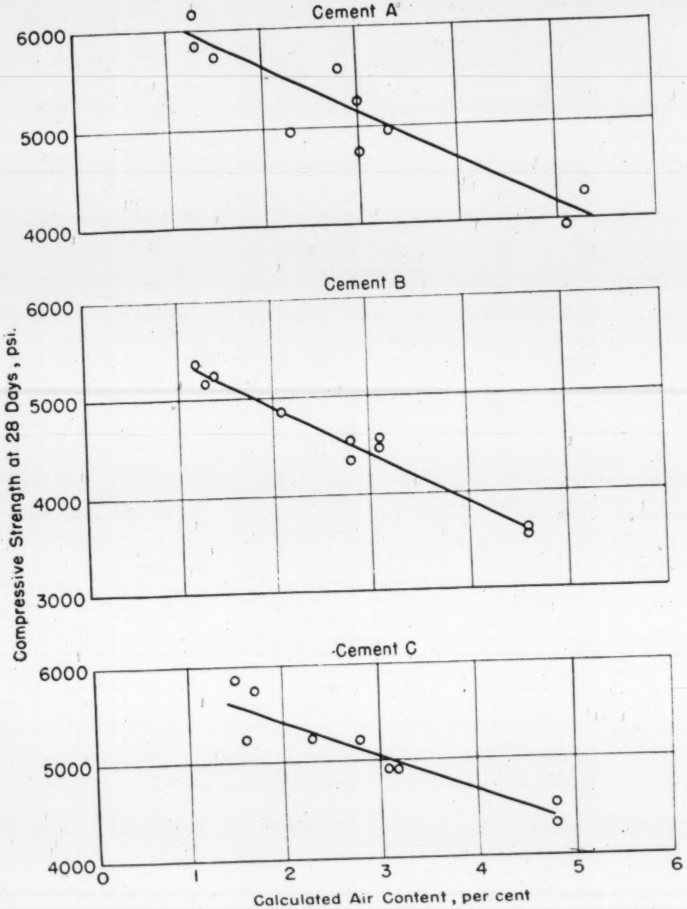

Fig. 1.—Relation Between Compressive Strength at 28 Days and Calculated Air Content—Series 1.

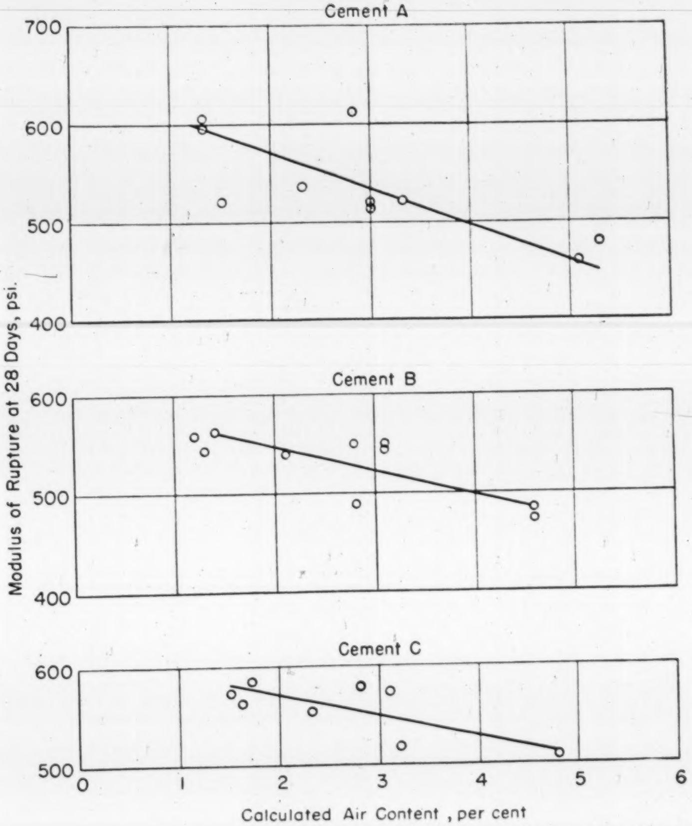

Fig. 2.—Relation Between Flexural Strength at 28 Days and Calculated Air Content—Series 1.

reciprocating motion of the screed. The screed was operated with its long axis parallel to the 4-ft. axis of the slab.

Following two passes of the screed, the slab was belted in a manner similar to that employed in actual road practice. The procedure described was carried out to simulate construction practice and induce bleeding in the slabs similar to that which frequently occurs in the field. The slabs were cured the first 24 hr. under wet burlap in the labora-

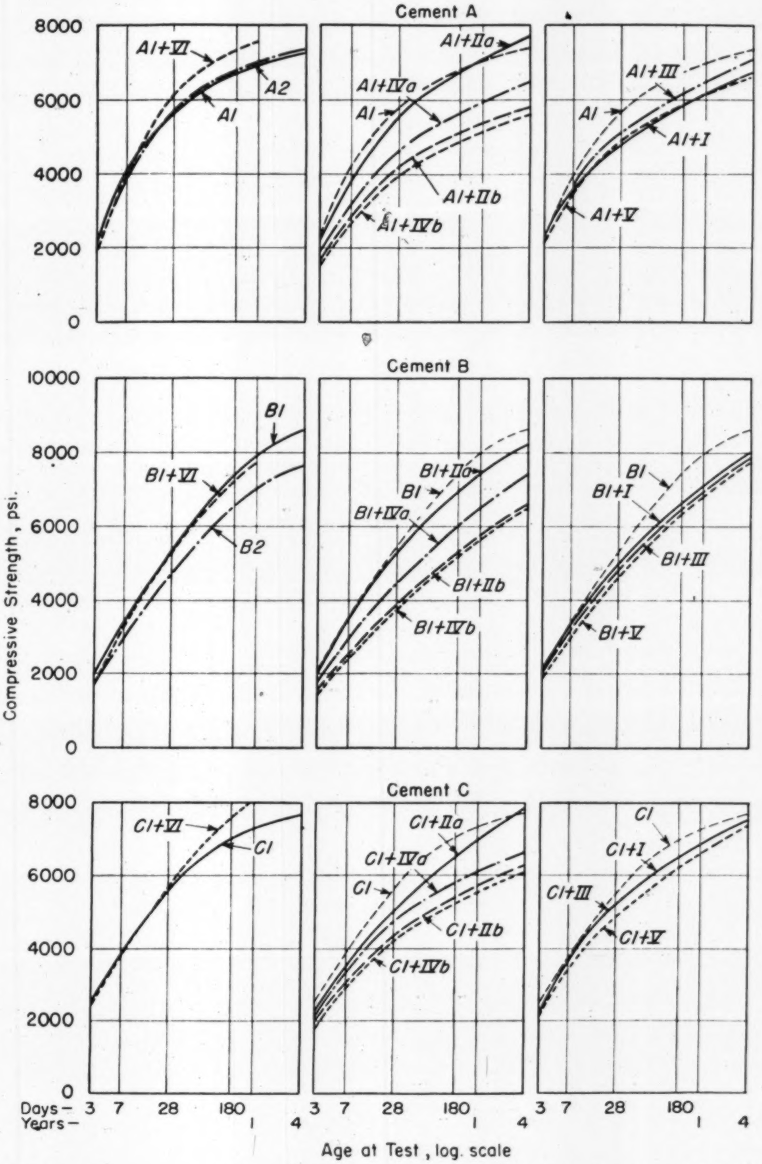

FIG. 3.—Compressive Strength of Concrete as Influenced by Cement—Series 1.

tory followed by one year's curing in the moist room. Five cores 6 in. in diameter were then drilled from each slab. These cores were returned to the moist room for another year of curing, after which 2-in. disks were cut from the tops and bottoms of three of the five cores from each slab. These disks were returned to the moist room for another 15 days' curing, after which they were air-dried at 70 F. and 50 per cent relative humidity, for seven

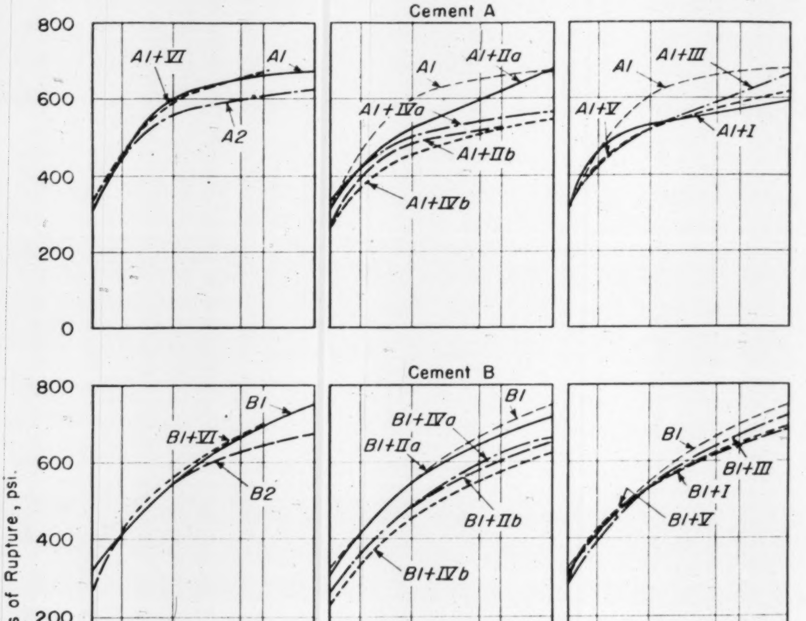

FIG. 4.—Flexural Strength of Concrete as Influenced by Cement—Series 1.

days. They were then immersed in a 10 per cent calcium chloride solution for 24 hr. before being subjected to alternate freezing and thawing in a 10 per cent calcium chloride solution. The disks were approximately 750 days old when freezing started.

The remaining two cores of the original five drilled from each slab were given approximately 15 months' additional moist curing, after which 2-in. disks were cut from the tops and bottoms of each core and tested for specific gravity and absorption. These disks were approximately 830 days old when tested.

DISCUSSION OF TEST RESULTS

Series 1:

Table III shows the calculated air contents for the different concretes. These varied from 1.2 per cent for concrete containing nonair-entraining cement B1 to 5.3 per cent for concrete containing blend A1 + IIb. All of the blends containing natural or slag cement produced air contents in excess of 2 per cent except natural cement IIa and slag cement VI. The air-entraining portland cements developed about 3 per cent air.

Results of compressive and flexural strength tests for specimens ranging in age from 3 days to 4 yr. are shown graphically in Figs. 1 to 4.

Figures 1 and 2 show the relation between air content and compressive and flexural strength at 28 days. The data at this age were selected for plotting as being typical of the data obtained at all ages. The test data indicate that, for air contents in excess of 2 per cent, the compressive strength was reduced approximately in proportion to the increase in the amount of entrained air. For the concrete with the two highest air contents, A1 + IIb and A1 + IVb, the compressive strength at 28 days was reduced approximately 30 per cent as compared to that obtained with plain portland cement concrete. The corresponding decrease at 4 yr. was about 24 per cent.

In Figs. 3 and 4 are plotted the age-strength relations for all cement combinations. In each case the data have been plotted in three groups. The left-hand diagrams show the age-strength relationships for the plain cement concretes, for the air-entraining portland cements (A and B only), and for the slag cement blends. The center diagrams show the corresponding values for concretes containing natural cements II and IV; that is, the two natural cements that had different amounts of interground air-entraining material. The right-hand diagrams give corresponding data for the other three natural cements.

TABLE IV.—MIX DATA—SERIES 2.[a, b]

Cement	Water content,[d] gal. per bag	W_c[e]	Slump, in.	Weight of Fresh Concrete, lb. per cu. ft.	Actual Cement Content,[d] bags per cu. yd.	Air Content	
						Calculated, per cent	Measured,[f] per cent
A1	5.8	0.169	3.5	147.8	5.8	0.9	1.7
A2	5.7	0.168	3.8	146.4	5.8	2.0	2.6
A1 + I	5.8	0.169	3.6	146.4	5.8	1.7	2.5
A1 + IIa	5.8	0.169	2.6	147.2	5.8	1.1	1.6
A1 + IIb	5.8	0.169	4.2	138.9	5.5	6.7	7.3
A1 + III	5.8	0.169	3.7	142.9	5.7	4.0	5.0
A1 + V	5.8	0.169	3.5	141.9	5.6	4.7	5.3
A1 + VI	5.8	0.169	3.2	147.0	5.8	1.2	2.1
B1	5.9	0.172	3.6	147.6	5.8	0.9	1.4
B2[g]	...	...	...	...	...	...	...
B1 + I	5.9	0.172	3.1	146.2	5.8	1.7	2.0
B1 + IIa	6.0	0.174	3.4	147.0	5.8	0.9	1.7
B1 + IIb	5.9	0.172	4.1	139.7	5.5	6.1	6.8
B1 + III	5.9	0.172	3.5	143.1	5.7	3.7	4.8
B1 + V	5.9	0.172	3.4	142.3	5.6	4.3	5.5
B1 + VI	6.0	0.174	3.4	147.0	5.8	0.9	1.8

[a] Mixes made about 2½ yr. after original mixes in series 2 to check air content. Cements same as used in original series.

[b] Mix by dry weight = 94:194:339, using 1-in. to No. 4 gravel.

[c] Where natural or slag cement was used, proportions were as follows: 86 per cent portland cement; 14 per cent natural or slag cement by weight.

[d] Where natural or slag cement was used, 94 lb. considered as one bag.

[e] Water per unit volume of concrete based on actual yield test.

[f] Using Pearson pycnometer.

[g] Cement B2 not available

An inspection of Fig. 3 reveals that: blends of all three plain portland cements with natural cements IIb and IVb showed considerable reduction in strength compared to the plain portland cement. With the exception of the slag cement VI, the other blends gave strengths intermediate between the plain portland and the two high air-entraining natural cements IIb and IVb. For several ages the blend A1 + IIa gave higher compressive strengths than the concrete made from plain portland cement. The percentage reduction in strength due to air entrainment was less, in all cases, at the 4-yr. period than at the 28-day period.

The combination of slag cement blend VI with each of the three portland cements showed approximately as high or higher strengths at one year than the portland cements with which it was blended. In general, the air-entrained portland cements showed slightly lower strengths than the companion plain portland cements. The reduction in strength is about what would be expected[4] based on the amount of calculated entrained air.

Blends of low air-entraining natural cements and portland cements gave markedly higher strengths than blends of high air-entraining natural cements and portland cements at all ages of test. This is plainly seen in the center diagram of Fig. 3.

These general relationships hold for all three of the portland cements used. The same trends brought out for com-

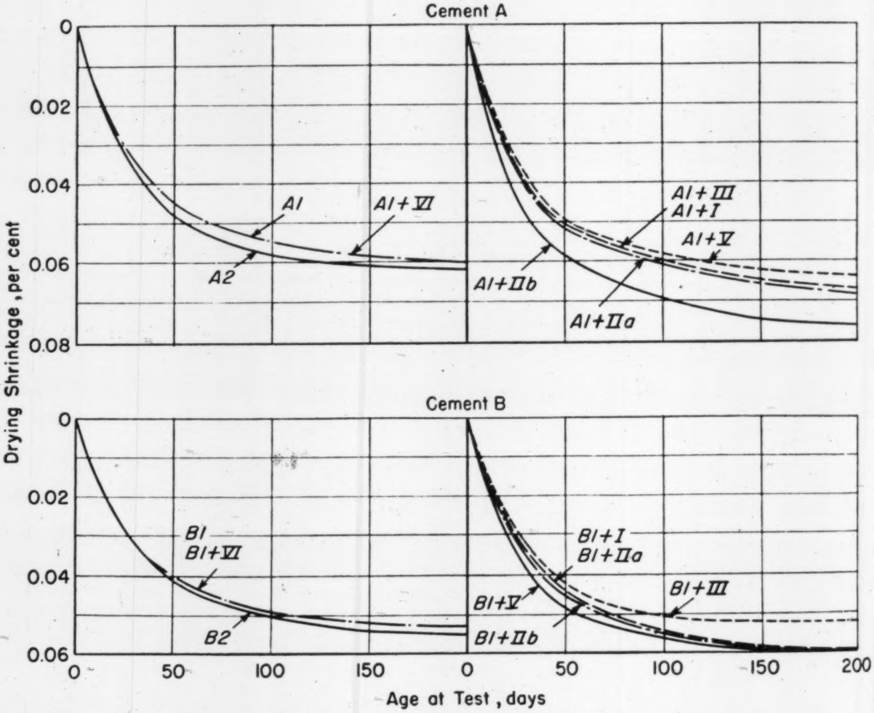

FIG. 5.—Drying Shrinkage of Concrete as Influenced by Cement—Series 2.

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[4] H. F. Gonnerman, "Tests of Concrete Containing Air-Entraining Portland Cements or Air-Entraining Materials Added to Batch at Mixer," *Journal*, Am. Concrete Inst., Vol. 15, No. 6, p. 477 (1944).

pressive strength are characteristic of flexure except that they are not so pronounced.

Series 2:

For each variable, seven 3 by 4 by 16-in. beams were made for test, three for drying shrinkage, two for freezing and thawing, and two for flexural strength tests as a standard of comparison for the specimens frozen and thawed. Mix data are shown in Table IV.

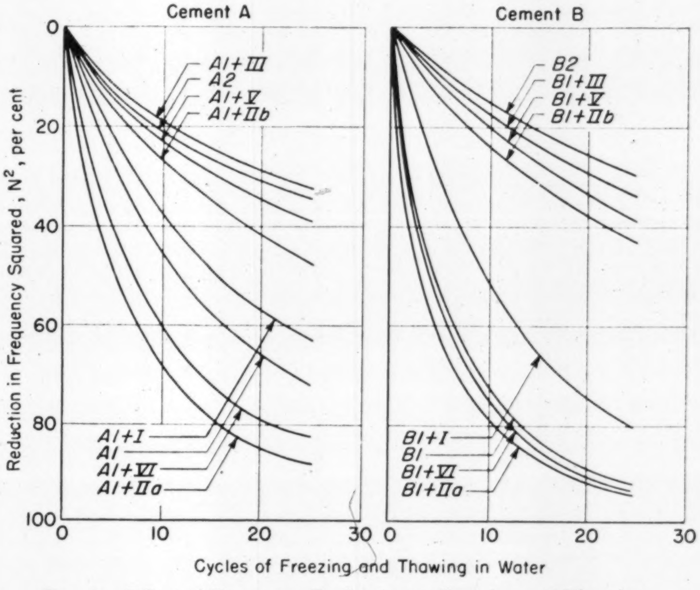

FIG. 6.—Effect of Cement on Resistance to Freezing and Thawing.

The initial reading of the drying shrinkage specimens was made after 24 hr. in the mold, under wet burlap. The specimens were then placed in air storage at 70 F. and 50 per cent relative humidity.

As may be seen in Fig. 5, there is no outstanding difference in the shrinkage of concrete made with any of the cements or blends tested. In the case of cement A1 blended with the air-entrained natural cement IIb, the curve indicates slightly greater shrinkage than the other cements tested. It is very doubtful that this small difference is significant. There is also a slight difference in the shrinkage characteristics of concrete containing cement A as compared to cement B but this may be a function of their composition. The principal difference in compound composition between the two cements is the tricalcium aluminate content, in which cement A is high (12 per cent) while cement B is low (6 per cent.)

The first group of freezing-and-thawing tests was made on 3 by 4 by 16-in. concrete beams cured continuously in moist air for 230 days before freezing started. The specimens were frozen and thawed immersed in water, the temperature range being from 70 to −20 F. The natural frequency of vibration of each specimen was determined sonically at 70 F. just before freezing and thawing started, and at the completion of each five cycles of freezing and thawing. The results of these tests are plotted in

Fig. 6. The test was discontinued at the end of 25 cycles because of the marked reduction in natural frequency exhibited by most of the specimens. The specimens were then tested as beams and the flexural strength determined. At the same time continuously moist-cured companion specimens that had not been frozen and thawed were tested, and the reduction in strength caused by freezing and thawing computed.

TABLE V.—
REDUCTION IN MODULUS OF RUPTURE AND FREQUENCY SQUARED OF 3 BY 4 BY 16-IN. BEAMS AFTER 25 CYCLES OF FREEZING AND THAWING IN WATER[a].—SERIES 2.

Cement	Modulus of Rupture, psi.[b]		Percentage Reduction in	
	Unfrozen specimens[c]	Frozen specimens	Modulus of Rupture	Frequency Squared[d]
A1	790	245	69	72
A2	770	525	32	35
A1 + I	730	285	61	66
A1 + IIa	705	130	82	88
A1 + IIb	695	295	58	48
A1 + III	720	430	40	33
A1 + V	720	395	45	40
A1 + VI	800	180	78	82
B1	780	110	86	92
B2	815	515	37	32
B1 + I	870	200	77	81
B1 + IIa	780	90	88	94
B1 + IIb	745	325	56	44
B1 + III	850	445	48	35
B1 + V	830[e]	440	47	39
B1 + VI	895	95	89	93

[a] Specimens stored continuously in moist air 230 days before freezing.

[b] Beams tested with center-point loading on a 14-in. span. Side of beams as molded in tension, 3-in. depth. Each value average of two tests unless otherwise noted.

[c] Specimens stored continuously in moist air 70 F. until tested. Tested at the same time freezing- and-thawing specimens were tested.

[d] Values are based on average of readings on 3 and 4-in. axes.

[e] One test only.

In Table V are given the flexural strength data and the percentage drop in sonic modulus of elasticity expressed in terms of the frequency squared (N^2). The values for modulus of rupture of the unfrozen specimens refer to the strength of the specimens that were stored in moist air continuously and tested at the same time the specimens subjected to alternate freezing and thawing were tested. These values are used in computing the values tabulated as "Reduction in Modulus of Rupture."

No unit weight determinations were made for the concrete mixes used in the series 2 tests and therefore it was not possible to compute the air contents of these mixes. In order to obtain data on their relative air contents a supplementary series of tests was made later, data from which are shown in Table IV. Comparing the calculated values for air content as shown in this table with the corresponding values for the mixes used in series 1 as shown in Table III it is found that in the cases of those combinations which can be classified as nonair-entraining, the values for series 2 are slightly lower than those for series 1. However, the differences average less than 0.5 per cent. In the case of the concretes that can be classed as air-entraining; those made with cement A2 and the combinations of A1 and B1 with natural cements IIb, III, and V, the values in series 2 are somewhat higher, the average difference being about 1.2 per cent. The one exception is air-entraining cement A2 which shows lower air content in series 2. Cement B2 was not available for the supplementary test in series 2.

Due to the fact that no determinations of air content were made on the concrete actually used in the freezing-and-thawing tests, only general reference to the relative air contents, as "high" or "low" is made in the discussion of these data. It is felt that the values reported in Tables III and IV are sufficiently consistent to warrant this classification.

Referring to the left-hand diagram in Fig. 6 (which gives the results for cement A), it is noted that there is a marked difference in resistance to freezing and thawing resulting from the use of different blends. The greatest reductions in N^2 (a measure of the breaking down of the structure of the concrete)[5] were found with blends containing natural cement IIa and the slag cement VI. The plain portland cement A1 gave results but slightly better. These concretes all had low air contents.

Greatly increased resistance was obtained with the air-entraining portland cement A2 and with the blends of A1 with natural cements IIb, III, and V, all of which gave relatively high air contents in concrete. The blends containing natural cement I gave intermediate results. The air content of these concretes could be classed as "intermediate."

In the right-hand diagram of Fig. 6 are shown similar curves for cement B. The essential difference between these results and those shown for cement A is the poorer resistance shown for the plain portland cement and the blends containing cements IIa and VI. In this case there is a greater spread between the concretes of poor resistance and those showing good resistance than with cement A. The results with blends of cement B1 confirm the conclusion from the results with cement A1 that the amount of entrained air is the governing factor between good and poor resistance to the action of freezing and thawing in water. The order of resistance using cement B1 was, with one exception, the same as that with cement A1.

The reduction in natural frequency squared, N^2, was accompanied by a corresponding reduction in flexural strength. This relation is shown in Fig. 7. The data indicate that the reduction in flexural strength can be predicted with reasonable accuracy from a determination of the reduction in N^2.

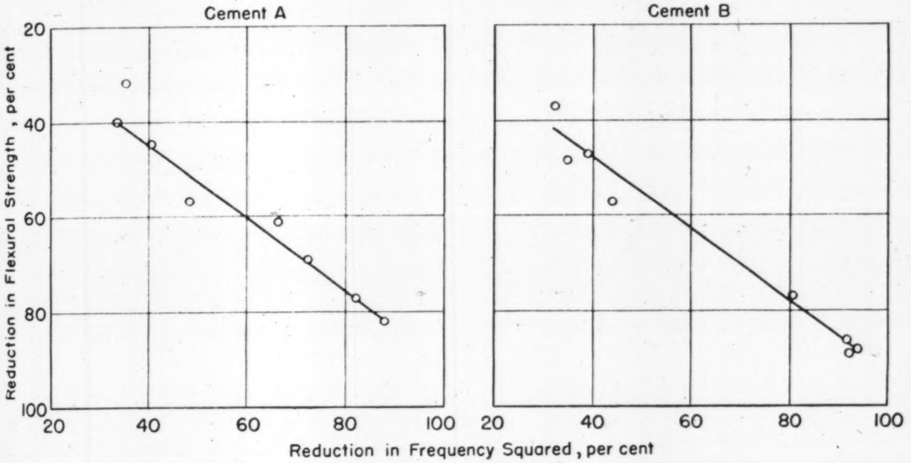

FIG. 7.—Relation Between Reduction in Flexural Strength and Reduction in Frequency Squared after 25 Cycles of Freezing and Thawing in Water—Series 2.

A second set of beam specimens in series 2 (same specimens used for shrinkage measurements) were cured under wet burlap for 24 hr., followed by storage in laboratory air at 70 F. and 50 per cent relative humidity for 260 days. They were then placed in water for 7 days, after which they were frozen and thawed in water, using the procedure previously described. At the

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[5] F. B. Hornibrook, "Application of Sonic Method to Freezing and Thawing Studies of Concrete," ASTM Bulletin, No. 101, December, 1939, p. 5.

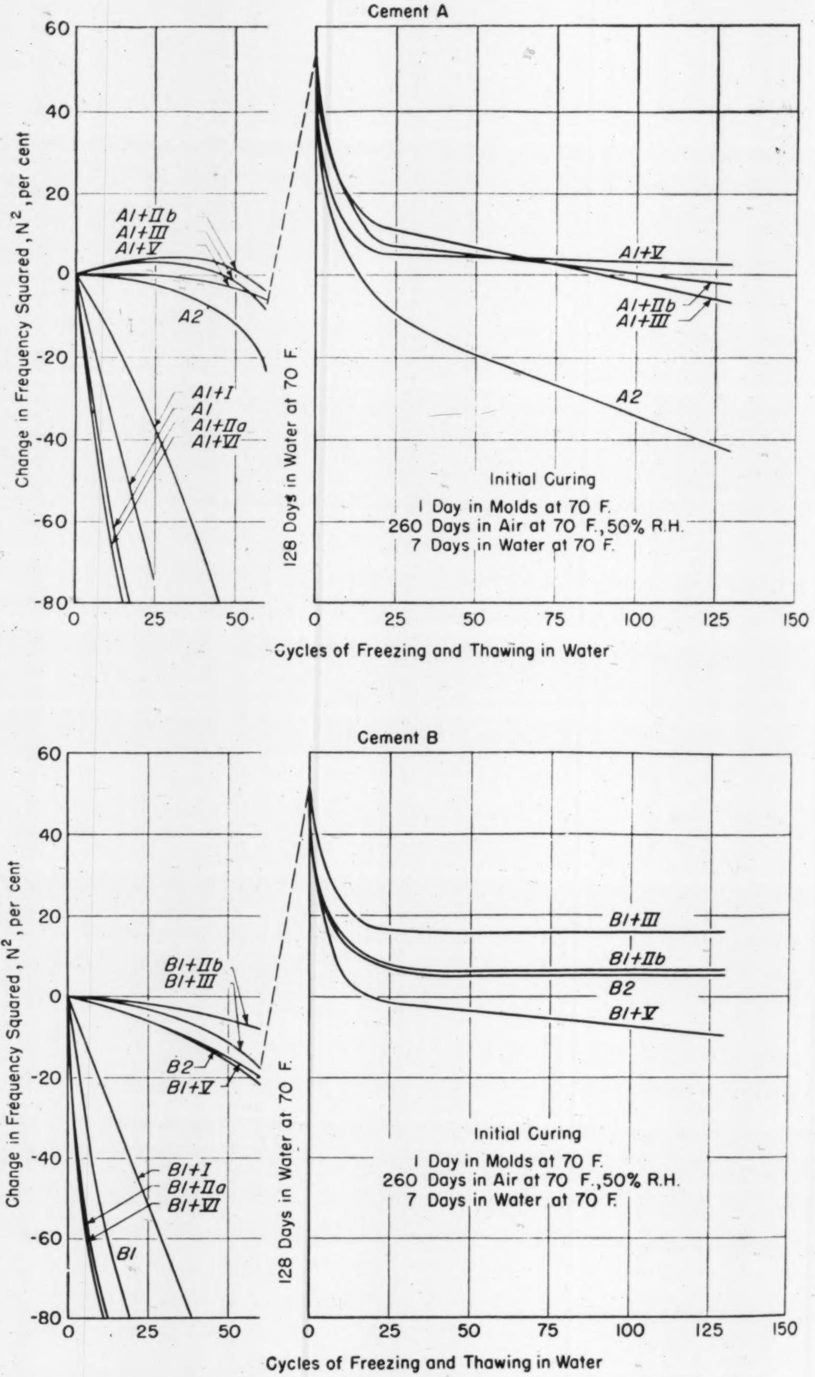

FIG. 8.—Effect of Cement on Resistance to Freezing and Thawing of Concrete.

end of 60 cycles of freezing and thawing, at which time many of the specimens had disintegrated, the test had to be discontinued temporarily because of other needs for the freezing equipment. Those specimens still remaining were stored in water at 70 F. for 128 days, after which they were again subjected to the freezing-and-thawing cycle. To simplify discussion, the first group of 60 cycles of freezing and thawing is referred to as the first phase and the second group of freezing-and-thawing cycles following the 128-day rest period as the second phase.

Results of these interrupted freezing-and-thawing tests are shown graphically in Fig. 8. These data for cement A in Fig. 8 indicate very rapid deterioration during the first phase for the plain portland cement A1 and the blends which had low air contents. Air-entraining cement A2 and the blends which had high air contents, however, showed greatly superior resistance by comparison to concrete of low air content. The combinations showing good resistance were the same as those showing good resistance in the tests made after continuous water curing (see Fig. 6). However, the shapes of the curves are reversed. The general trend at 60 cycles was for a rapid drop in N^2. As previously explained, those combinations which had not failed at 60 cycles were stored in water 128 days. At the end of this period they recovered the loss in N^2 indicated at 60 cycles and gained about 45 per cent over the initial amount indicated before the first phase of freezing and thawing. As may be seen from the curves for the second phase, the rate in reduction of N^2 was very rapid for the first 20 cycles of freezing and thawing but thereafter with the exception of the curve for air-entraining cement A2 the rate was relatively slow. At 145 cycles of freezing and thawing, the value of N^2 for the three blends was about the same as that obtained at the start of the first phase of the test.

The shapes of the curves for the second phase are not the same as those for the first phase but are more like those shown in Fig. 6. Apparently the resistance to deterioration is influenced by the curing and the moisture condition of the specimens at the time freezing and thawing is started which accounts for the difference in the shape of the curves. The curves for the second phase in Fig. 8 and also those shown in Fig. 6 represent concretes that had been subjected to prolonged moist curing. Conversely, the results for the first phase in Fig. 8 are for concretes exposed to the drying action of air at 70 F. and 50 per cent relative humidity over a long period of time.

It has been shown by other investigations that specimens which have been subjected to cycles of freezing and thawing will, when given a rest period in water, show a recovery in sonic modulus of elasticity.[6]

Weight determinations made during the first phase indicated that all the specimens absorbed water during the first 10 cycles of freezing and thawing and some continued to gain in weight up to 40 cycles. Those specimens that gained the least in weight were those which showed low air contents. They also broke down rapidly in the freezing-and-thawing tests. Those specimens that gained the most in weight over a longer period were those of relatively high air contents. These gave good resistance in the freezing test. The results indicate that those concretes of low air content were practically saturated after a 7-day immersion period. The concretes having relatively high air contents were not completely saturated at the end of the 7-day immersion period and continued to absorb water during the freezing-and-thawing cycle.

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[6] Progress Report, Committee on Durability of Concrete, *Proceedings*, Highway Research Board, Vol. 24, p. 196 (1944). See Fig. 17.

The foregoing observations are offered as an explanation of the shape of the curves for the concretes showing good resistance in the freezing-and-thawing test during the first phase rather than as an explanation of the difference in behavior between air-entrained and non-air-entrained concrete.

Results shown in Fig. 8 for blends with cement B parallel very closely those shown for cement A. The plain cement B1 and those blends of low air content gave very poor resistance, the order of resistance being exactly the same as with cement A. Concretes showing good resistance at the end of 60 cycles of the first phase also exhibited about the same increase in N^2 during the 128-day water-storage period as was shown for the corresponding blends of cement A in Fig. 8.

TABLE VI.—SPECIFIC GRAVITY AND ABSORPTION OF 2-IN. DISKS CUT FROM TOP AND BOTTOM OF CORES 6 IN. IN DIAMETER AND 8 IN. IN HEIGHT—SERIES 3.

Cement	Specific Gravity,[a] 830 days' Moist Curing[b]		Absorption,[a] 830 days' Moist Curing,[c] per cent		Specific Gravity[a] (Dry)[d]		Absorption,[a] 5 hr. Boiling,[e] per cent	
	Top	Bottom	Top	Bottom	Top	Bottom	Top	Bottom
A1	2.43	2.47	5.06	4.27	2.33	2.37	4.94	4.22
A2	2.36	2.42	5.90	4.56	2.23	2.32	6.39	4.93
A1 + I	2.41	2.45	5.38	4.50	2.29	2.34	5.60	4.76
A1 + IIa	2.42	2.46	5.54	4.62	2.30	2.35	5.48	4.62
A1 + IIb	2.33	2.38	5.85	4.58	2.20	2.27	7.01	5.66
A1 + III	2.35	2.41	6.18	4.56	2.22	2.31	6.88	5.26
A1 + IVa	2.38	2.43	6.27	4.84	2.24	2.32	6.52	5.15
A1 + IVb	2.32	2.37	6.10	4.76	2.18	2.27	7.50	5.64
A1 + V	2.36	2.41	5.96	4.83	2.22	2.30	6.80	5.38
A1 + VI	2.42	2.46	5.71	4.45	2.29	2.36	5.65	4.39
B1	2.44	2.50	5.56	4.08	2.31	2.40	5.38	4.03
B2	2.34	2.42	5.64	4.34	2.21	2.32	6.74	4.96
B1 + I	2.39	2.45	5.96	4.35	2.26	2.35	6.32	4.62
B1 + IIa	2.41	2.46	5.98	4.66	2.27	2.35	5.88	4.52
B1 + IIb	2.34	2.39	5.76	4.73	2.21	2.29	6.80	5.57
B1 + III	2.36	2.44	6.22	4.70	2.22	2.33	6.66	4.90
B1 + IVa	2.38	2.44	6.34	4.88	2.24	2.32	6.45	4.92
B1 + IVb	2.33	2.41	6.38	4.38	2.19	2.31	7.27	5.10
B1 + V	2.35	2.44	6.39	4.39	2.21	2.34	6.98	4.96
B1 + VI	2.42	2.48	5.88	4.54	2.29	2.37	5.82	4.38

[a] Each value average of two tests
[b] Bulk specific gravity based on the wet. weight after 830 days in moist air.
[c] Based on the wet weight after 830 days in moist air.
[d] Bulk specific gravity based on weight after drying to constant weight at 350 F.
[e] Based on weight after 5-hr. boiling and 19 hr. in water.

The data show that interrupting the daily cycle of freezing and thawing and allowing the test specimens to remain unfrozen in water will result in a recovery in sonic modulus.

Series 3:

In this series, 6-in. cores were drilled from the 2-ft. square by 8-in. thick slabs. Two-inch disks were sawed from the tops and bottoms of the cores. In order to determine the difference in quality resulting from the use of the various combinations, the disks were tested for absorption and specific gravity. Upon removal from moist storage they were dried to constant weight at 350 F. They were then boiled for 5 hr. and allowed to stand in water for 19 additional hours, after which they were again weighed. From these data, absorptions and specific gravities were calculated by two different methods: First, on the basis of weights resulting from curing for 830 days in moist air; and second, on the basis of weights after boiling for 5 hr. followed by immersion for 19 hr.

From the results given in Table VI it is readily apparent that the disks from the top showed higher absorption than those from the bottom of the slabs. This was true irrespective of whether plain portland cement, air-entraining portland cement, or blended cement was used. The disks representing air-entraining concrete showed nearly as great a difference between top and bottom as those representing concrete in which the amount of entrained air was low.

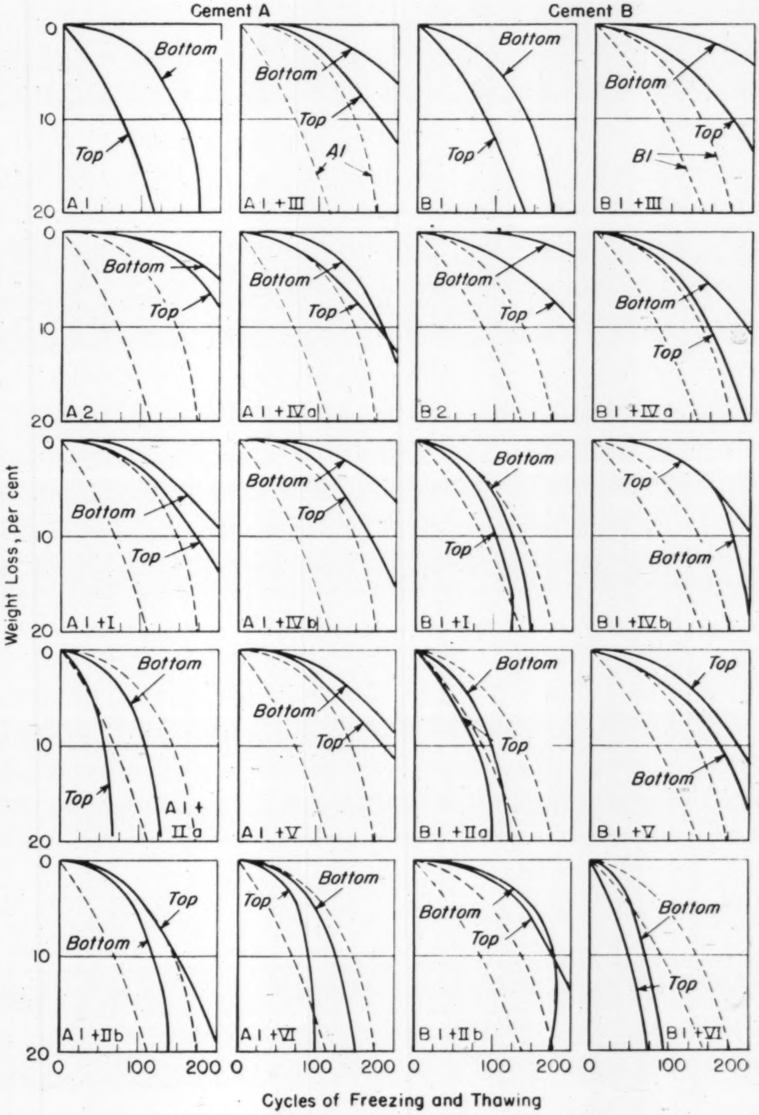

FIG. 9.—Weight Losses of Disks Cut from Tops and Bottoms of Concrete Cores Frozen and Thawed in 10 per cent $CaCl_2$ Solution.

The use of air-entraining concrete did affect the ratio of absorption after boiling for 5 hr. to absorption due to moist curing for 830 days. Considering the plain portland cements and the blends containing natural cements IIa, IVa, and VI, it will be noted that approximately the same values were obtained for absorption in both tests. The above combinations all had low air contents. The blends containing cements IIb and IVb gave much higher values for absorption in the 5-hr. boiling test than in the 830-day moist curing test. These two blends showed the highest air contents of any concrete tested.

The weight losses after freezing and thawing of 2-in. concrete disks cut from the tops and bottoms of the cores are shown graphically in Fig. 9.

In general, the bottoms of the cores showed more resistance to freezing and thawing in calcium chloride solution than the tops. There were only three exceptions out of 20 cement combinations. The plain portland cement concretes A1 and B1 and the blends A1 + IIa, A1 + IIb, A1 + VI, B1 + I, B1 + IIa and B1 + VI, all showed relatively poor resistance to freezing and thawing in calcium chloride solution. Four of the blends, A1 + IIa, A1 + VI, B1 + IIa, and B1 + VI, had poorer resistance than the comparable plain portland cements, whereas the air-entraining portland cements and the blends of higher air content were of benefit in producing better resistance than was furnished by the plain cement. This confirms the conclusion based on freezing-and-thawing tests of beam specimens in water. The improvement in resistance over the plain portland cement concrete was generally more pronounced for the tops than for the bottoms of the cores.

Summary

The following summarizes the test results:

1. Blends containing the nonair-entraining natural cements had higher strengths than blends of air-entraining natural cements at all ages of test.

2. For all combinations the strengths of the concretes were reduced as the air contents increased. There was a straight line relationship between strength and air content.

3. The blends of the slag cement with each of the three portland cements had equal or higher strengths at one year than the same portland cements.

4. There were no outstanding differences in the drying shrinkage characteristics of concretes made with any of the cement combinations tested.

5. Greatly increased resistance to freezing and thawing in water was obtained with the air-entraining portland cements and with the blends which produced relatively high air contents in concrete.

6. In the interrupted cycles of freezing and thawing in water, only those concretes having relatively high air contents had sufficient resistance to survive the first phase of 60 cycles as indicated by a drop in N^2 of less than 25 per cent. After a further storage period of 128 days in water these same high air concretes developed further resistance as indicated by a recovery of N^2 which surpassed the initial reading at the start of the first phase. In the subsequent freezing and thawing, or second phase, they had better resistance at 145 cycles than they did at 60 cycles in the first phase.

7. Under continued moist curing the concrete with the low air content was more nearly saturated than was the concrete with the high air content. When these concretes were dried and boiled, the concrete with the high air content absorbed more water than it did under moist curing, whereas the concrete with the low air content did not.

8. In practically all cases, the bottoms

of the cores gave better resistance to freezing and thawing in calcium chloride solution than the tops. The air-entraining portland cements and in general the natural cement blends gave better resistance than the plain portland cements.

CONCLUSIONS

These tests confirm the results of previous investigations to the effect that increased resistance to freezing and thawing is obtained by the use of air-entraining concrete.

It is concluded that the increased resistance to freezing and thawing of concretes containing blends of portland cement and certain natural cements, in the proportions used in this investigation, is due to the fact that the concrete produced with these blends contained entrained air.

DISCUSSION

MR. IRA PAUL[1] (*presented in written form*).—The data reported in this paper on "The Effect of Blends of Natural Cement on Properties of Concrete" are entirely based on laboratory fabricated specimens. Now let us look at the record of these blends in the field and compare the laboratory test results with actual service.

The standard practice of the New York State Department of Public Works in all concrete construction either for pavement or structure calls for the use of the natural cement blend with portland, in the proportion of about 14 per cent natural cement with 86 per cent portland cement by weight.

Since 1938 the New York State Department of Public Works has constructed 284 concrete paving projects with the blends of natural cement and portland cement covering a total of over 900 miles in addition to 13 experimental pavements during 1935–1936. Concrete cores removed from these pavements were tested in the laboratory for freezing and thawing in a 10 per cent calcium chloride solution to determine the resistence to salt action used in ice control. Field inspection of the pavements made subsequently, in general checked the laboratory test results obtained on the pavement cores. It was found that certain types of natural cement blends either reduced or eliminated the scaling due to the application of chloride salts. Those interested in the details of our findings should consult my paper[2] on "Chloride Salts-Resistant Concrete in Pavements" presented before the Association of Highway Officials of the North Atlantic States in February, 1938.

In order to bring these data up to date in the light of the paper presented by the Public Roads Administration both laboratory as well as field tests for air entrainment of the blended cement concrete were conducted on several experimental and regular pavement projects.

The natural cement which we used on over 98 per cent of our concrete projects and are still using requires a chemical analysis of CaO not to exceed 50 per cent, and MgO content not less than 15 nor more than 25 per cent, and ignition loss not in excess of 10 per cent. Beef-tallow content in the natural cement must be between 0.05 and 0.1 per cent. An hydration test is specified to control the degree of burning of this natural cement. Another type of natural cement is permitted in which beef tallow in an amount of not less than 0.2 nor more than 0.3 per cent by weight may be used. The latter type also contains about 5 per cent portland cement, which is added in order to meet the tensile strength requirement of neat briquet specimens of 125 psi. in 7 days and 200 psi. in 28 days. No portland cement is added at the mill to former type of natural. Actually the bulk of the type of natural cement we have used for the past twelve years more or less conforms with the chemical and physical properties of Natural Cement I listed under Tables II and IV in the paper under discussion, except that the autoclave expansion test is much higher, the specific gravity lower, and the tallow content between 0.05 and 0.1 per cent.

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[1] Director, New York State Department of Public Works Laboratory, Albany, N. Y.

[2] Ira Paul, "Chloride Salts-Resistant Concrete in Pavements," *Proceedings*, Assoc. Highway Officials of the North Atlantic States, Feb., 1938, p. 144.

When the A.S.T.M. Tentative Method of Test for Air Content of Portland Cement Mortar (C 185 – 46 T)[3] was first proposed, I had occasion to test the air content of the portland-cement mortars and blends with natural cement of the type used in 98 per cent of our construction work. A portland cement which showed only 7.3 per cent air in the mortar test gave an air content of 14.5 per cent in the natural cement blend with the same portland cement in the proportion of 14 per cent natural and 86 per cent portland. Subsequently, concrete cylinders were cast with the straight portland and the natural cement blend. Actual weights of the concrete in a 0.5-cu. ft. calibrated container showed the natural cement blend to contain only 1 per cent air. The hardened concrete cylinders were then subjected to freezing-and-thawing tests in a 10 per cent calcium chloride solution and it was found that the blended cement concrete lasted 100 cycles, whereas the straight portland only 71 cycles before complete disintegration. These and other laboratory tests were made with similar results. The next step was to check the question of air content on concrete placed in pavement under field conditions. Again the natural cement blend with the same portland cement tested in the laboratory showed a loss in weight with a 0.5-cu. ft. measure equivalent to 0.7 per cent air. In another experimental section of the same pavement 14 per cent of Daragg was substituted in place of the natural cement. Daragg is a finely pulverized, water-cooled slag to which 0.2 per cent Darex was added. The loss in weight per cubic foot of concrete with the Daragg replacement was also equivalent to 0.7 per cent air. Cores removed from the pavement when 3 months old showed excellent resistance to freezing and thawing at 100 cycles in a 10 per cent calcium chloride solution in spite of the low air content in the concrete. However the weight losses on pavement cores in the freezing-and-thawing tests did not check the tests on laboratory fabricated specimens with the same materials. The natural cement blend on the laboratory-fabricated specimens when subjected to freezing-and-thawing tests in a 10 per cent $CaCl_2$ solution completely disintegrated at 100 cycles, whereas the cores from the pavement at the same 100 cycles test showed a loss of only 5.1 per cent.

TABLE VII.—COMPARISON BETWEEN PAVEMENT CORES AND LABORATORY CORES IN FREEZING-AND-THAWING TESTS IN 10 PER CENT CALCIUM CHLORIDE SOLUTION.

Cement	Weight Loss, per cent							
	Field Cores from Pavement				Laboratory Cores from 24 by 24 by 8-in. Block			
Number of Freezing-and-Thawing Cycles...........	25	50	75	100	25	50	75	100
Portland No. 1.......	0.0	2.1	4.0	8.7	0.5	3.1	5.8	33.9
Portland No. 1 + tallow............	0.0	0.0	0.0	2.1	0.3	3.9	31.2	61.4
Portland No. 1 + natural............	0.0	0.1	2.3	6.8	2.4	24.9	100	...
Portland No. 2.......	0.0	0.0	1.6	6.3	1.6	12.3	62.5	100
Portland No. 2 + tallow............	0.0	0.0	0.0	1.7	0.1	7.0	15.0	46.8
Portland No. 2 + natural............	0.0	0.0	0.5	4.3	1.5	4.3	27.6	83.8

We have noticed similar lack of correlation between laboratory fabricated specimens and field specimens removed from pavements or structures. A few years ago I fabricated several concrete blocks, whose dimensions were 24 by 24 by 8 in., from the same materials and with the same proportions used in a concrete pavement. The blocks were manipulated in exactly the same manner as the pavement itself. Cores were drilled from these blocks and compared with the cores removed from pavement for the freezing-and-thawing tests. No air-entrainment determinations were recorded. Two portland cements, two tallow-treated portland cements, and two natural cement blends with the untreated portland cements were used with the same aggregates. The results are shown in the accompanying Table VII.

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[3] 1946 Book of A.S.T.M. Standards, Part II, p. 1277.

In Table VII, please note that there is no comparison in test results between the cores from pavement and those from the laboratory-fabricated specimens. A recent inspection of this pavement, which is nine years old, shows all sections in which these six cements were used in good condition.

In the construction of the Idlewild Airport concrete runways, New York State Department of Public Works Specifications on the blend of natural and portland cements are being used. Air-entrainment determinations by the gravimetric method in accordance with A.S.T.M. Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138 – 44)[4] were made on the fresh concrete. The materials engineer who made these tests reported about 1.4 per cent air in the natural cement blend concrete. Freezing-and-thawing tests in a 10 per cent calcium chloride of cores drilled from various sections of the 12-in. thick concrete showed the same good results as the cores removed from State work.

My observations and investigations on the natural cement blend as well as the so called air-entrained portland cements have led me to believe that the air content in concrete is not the answer. What is really formed in the concrete with additions of tallow, fish oil, vinsol resin, or Darex, is a waterproofing agent. When neutralized Vinsol resin, which is a soap known as sodium resinate comes in contact with the lime water liberated in the hydration of the cement, an insoluble colloidal precipitate of calcium resinate is formed. All the particles of aggregate are coated with the colloidal calcium resinate which is insoluble in water, sodium chloride, and calcium chloride solutions. It is this waterproofing agent that does the trick, and prevents the penetration of the chloride salts into the pores of the concrete. In the cases of flake unneutralized vinsol, fish oil, or tallow added to the cementitious material, these unsaponified agents must first be neutralized by the alkalies liberated in the cement during its hydration. That portion of the agent which is neutralized will then react with the lime in solution.

During this chemical reaction between the so-called air-entraining agent, cement and water air bubbles are liberated and are visible to any engineer who has used this material. Various means and apparatus are being studied to measure the air content of the concrete. There are too many variable factors which will affect the air entrainment determinations in concrete, any one of which or a combination of these will change the results obtained. Among these variables are the type of aggregates, the gradation of the aggregates, type of portland cement, water-cement ratio, amount and type of air-entraining agent, length of mixing time of concrete and manipulation of the concrete, and many others. Such being the case we prefer specifying the *quality* of the portland and the natural cement for the blends with appropriate controls on these cementitious materials in accordance with New York State Specification requirements. The kind and the amount of the particular so-called air-entraining agent is added to the material at the cement mill in accordance with our specification requirements. In this way we are assured of getting comparable satisfactory results regardless of what cement mill manufactures the material, with no air-entrainment control at the concrete mixer.

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[4] *Ibid.*, p. 513.

Such tests as we have made in the field during actual construction to determine the percentage of air entrained in the natural cement blend with portland meeting our Department specifications have been in the low ranges of approximately 1 per cent. Cores tested for durability in the laboratory from those pavements and structures in which the natural cement and portland cements did meet our State Specification requirements have rendered satisfactory service in the field.

Finally, I should like to say one more word about the waterproofing characteristics of the so-called air-entraining agents. Additives such as vinsol resin, tallow, fish oil, and Darex to cementitious materials produce an integral waterproofing agent in the concrete mix, which is due to the chemical reaction between these agents and the hydration products of the cement. On the other hand, direct waterproofing agents such as road oils, or asphalt emulsions incorporated in non air-entrained portland cement mix will produce equally durable concrete as that in which the integral waterproofing agent is formed.

MR. W. C. VOSS.[5]—I should like to ask Mr. Paul whether the block he talked about, because of the tremendous difference in the resistance to freezing and thawing, was cast upon as absorbent a base as the pavement was.

MR. PAUL.—This block of concrete I described was cast on the laboratory floor on paper.

MR. J. H. SWANBERG.[6]—We are particularly interested in this paper because during the past six years we have done considerable work in our own laboratory with blends of natural and portland cement. However, we are unable to evaluate the effect of the natural cement without air entrainment, because in our laboratory tests of freezing and thawing, the air contents have ranged from 3.4 to 4.8 per cent. Concrete produced from natural cement under field conditions of mixing has resulted in lower air contents, but the concrete cannot be directly evaluated for durability. In tests of cores drilled from six different paving projects at age 2 yr., we found the compressive strength of blended cement concrete to be 84.2 per cent of the strength of straight portland-cement concrete. In laboratory tests using two sources of natural cement and keeping constant the cement-voids ratio, we found practically identical strengths at age 90 days for blended and for straight portland-cement concrete. This applied to both compressive and flexural strengths.

Of particular interest, in the author's paper, is the very marked recovery exhibited by freezing-and-thawing specimens during the 128-day period in water at 70 F. This serves to illustrate why the 40 F. thawing water sometimes produces a more rapid destruction in freezing-and-thawing tests.

Also of considerable interest is the fact that the beneficial effect of a preliminary period of drying in air, as shown in Fig. 8 as compared with Fig. 6, is largely confined to samples of high air content. This indicates that a period of drying in air is necessary in order to obtain the full benefit from air entrainment.

It is unfortunate that the air content was not determined for the concrete upon which freezing-and-thawing tests were made. However, the supporting data indicate that the air content is known within reasonable limits. A study of Fig. 9 indicates that the resistance to freezing and thawing in a calcium chloride solution is not closely related to the air content. Several years ago we read in an abstract of some tests performed in Stockholm that a $3\frac{1}{2}$ per cent solution of calcium chloride was found to be the most destructive in tests of this kind. While we suspect that the exact percentage to be used must be related to the freezing temperatures employed, we nevertheless found a very rapid destruction when using a $3\frac{1}{2}$ per cent solution. Standard portland-cement concrete disintegrated completely at 35 cycles, while blended and air-entrained concretes had lost from 2 to 6 per cent of their weight. At 122 cycles the loss in weight ranged from 30 to 90 per cent for blended and air-entrained concrete and was rather well related to the air content.

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[5] Head, Department of Building Engineering and Construction, Massachusetts Institute of Technology, Cambridge, Mass.

[6] Engineer of Materials and Research, Minnesota Department of Highways, Minneapolis, Minn.

MR. ELLIOT A. HALLER.[7]—Our most recent experience with natural cement that is pertinent to this paper and to Mr. Paul's discussion has been in connection with tests made by us of the concrete from the New York Municipal Airport at Idlewild. A blend of portland cement and natural cement produced results that were extremely high in compressive strength, flexural strength, and resistance to alternate freezing and thawing without entraining amounts of air that were any greater than the amount entrained in normal portland-cement concrete mixtures.

MR. LEO KAMPF.[8]—The Idlewild Airport was constructed in our Borough, so I have had a little opportunity to observe it.

In the first place, the maximum age of the runways is about three years—hardly enough time to judge how well it has stood up under freezing-and-thawing conditions. Furthermore, the airport has not yet been put into operation.

The second factor is that the slump of the concrete put into the runways rarely exceeded 2 in. Those of us who have run freezing-and-thawing tests know that this is an important factor in resistance to freezing and thawing. Obviously, with 2-in. slumps the concrete could stand up a lot better than it would with the higher slump that is frequently used.

Mr. Paul stated that the losses of the laboratory-prepared specimens were much higher than the field losses. There, too, it is a question of technique. We all know that wet aggregates cause concrete to fail more rapidly under freezing and thawing. Aggregates in the field are usually, though not always, in a wet condition. Now, I do not know whether Mr. Paul's laboratory-prepared specimens were made with wet aggregates or whether his laboratory prepared specimens were worked more than the field specimens, all of which could account for the differences.

Furthermore, apparently his tests were based upon the loss in weight which is not too good a measure of resistance to freezing and thawing. Many of us have experienced specimens of concrete put through freezing-and-thawing tests at the end of a hundred cycles or so that show no loss and yet have considerable reductions in flexure and other measurable, tests of resistance to freezing and thawing.

Observations in the field and in laboratory tests indicate that freezing and thawing can manifest itself in one or both of two ways. It can show up as spalling or as cracking. Spalling can be measured by the loss in weight. It occurs, however, only when excessive water has been used in the concrete, when the expansive force due to a large amount of water freezing becomes great enough to cause destruction. Cracking is caused by a reduction in the modulus of rupture resulting from internal stress of a lesser degree. It occurs in the field when the flexural strength of the concrete is reduced below the flexural loads encountered. It would not occur, therefore, in concrete whose modulus of rupture is higher than the flexural loads placed on it.

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[7] Vice-President, The Haller Testing Laboratories, Inc., New York, N. Y.

[8] Chemist in Charge, Div. of Analyzing and Testing, Borough of Queens, City of New York, Kew Gardens, N. Y.

It is, therefore, apparent that the loss in weight cannot be depended upon as a measure of the resistance to freezing and thawing of concrete.

MR. H. F. GONNERMAN.[9]—This paper adds considerable additional confirmatory information to our knowledge of the properties of concrete made with non air-entraining and with air-entraining portland cements and blends of the former with different natural cements ground both with and without air-entraining agents. The results of strength, shrinkage, and freezing-and-thawing tests presented in the paper are in accord with those from tests made along similar lines by other investigators.

The authors attribute the increased resistance to freezing and thawing of the concretes containing blends of portland cement and certain natural cements to the additional air entrained in the concrete with these blends. This conclusion is supportd by freezing-and-thawing tests made in our Research Laboratories on concrete specimens containing blends of portland and natural cement. In these tests natural cements ground both with and without air-entraining agents were blended with a non air-entraining portland cement (86 per cent portland, 14 per cent natural cement by weight). In the case of the blends of natural cements ground without the air-entraining agents, the resistance of the concrete to freezing and thawing was comparable to that of the concrete made with the non air-entraining portland cement. However, when the natural cements ground with the air-entraining agents were used in the blends, the air content of the concrete was increased a substantial amount and also the resistance to freezing and thawing, just as in the tests reported by Messrs. Timms, Grieb, and Werner. The results of these laboratory tests are in agreement with published tests of cores taken from concrete pavements constructed with various blends of portland and natural cement.[10]

MR. BERTRAND H. WAIT[11] (*by letter*).—I have read and studied Mr. Timms' paper with a great deal of interest.

It would appear that the conclusions in this report should go much further than to state that the increased resistance to freezing and thawing was due to the fact that the concrete contained entrained air in approximately the amounts now specified by the A.S.T.M. for air-entrained concrete.

Engineers who have studied the production of concrete resistant to freezing and thawing know that the resistance depends largely on the quality of the portland cement, the quality of the natural cement, the quality of the grinding compound or waterproofing used and the method of incorporating the waterproofing into the cement or concrete. Hundreds of tests and hundreds of miles of concrete road in service in New York State have proved conclusively over a period of twelve years that resistant concrete can be obtained without high loss of strength and without high waterproofing or air entrained content. The data on these tests have been published to some extent and are all available to any one wishing to make a thorough investigation of the subject.

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[9] Director of Research, Portland Cement Association, Chicago, Ill.

[10] C. W. Allen and J. F. Barbee, "Ohio's Experience with Vibratory Methods of Placing Concrete and Tests with Grinding Aids," *Proceedings*, Highway Research Board, Vol., 22 p. 306 (1942).

[11] President, The Wait Associates, Inc., New York, N. Y.

In the field over a period of thirty years, hundreds of miles of concrete road laid with a satisfactory portland cement with very small air entrainment have proved satisfactory in resistance to scaling, while hundreds of miles laid with other portland cements which were not as good have failed.

During the past twelve years New York State has obtained excellent results and on the average at least equal to any other concete ever laid, by using a blend of specially prepared portland cement together with a natural cement furnished under a specification which required a natural cement rock which was uniform and met strict chemical requirements. In all of this work the state has specified tallow grinding compound to be added to the natural cement in very small quantities and that one bag of the natural cement per cubic yard of concrete be used as a replacement for one bag of the non air-entraining Portland cement.

Also, I would refer to a report by Mr. Wagner at the 1940 Spring Meeting of Committee C-1 in the "Symposium on Specifications for and Additions to Portland Cement"[12] which shows clearly that higher resistance to freezing and thawing with slight loss in strength was obtained with waterproofing similar to the tallow specified by New York State for use in portland-natural blends.

An article by Mr. M. A. Swazey published in *Engineering News Record* June 9, 1941 reports excellent results in freezing and thawing with a small amount of crusher oil present in cement Type 2 with additional loss in weight of only 0.7 lb. per cu. ft. or less than ½ per cent increase in weight loss. Compressive strengths at all periods were equal to the straight portland cements, when six bags of cement per cubic yard were used, which is near the average for highway concrete.

The Kellerman and Runner (Public Roads Administration) report presented at the 1938 Annual Meeting of the A.S.T.M. covered a general test series on portland and portland-natural blends. The results of these tests with portland cement and the natural cement specified in New York State closely parallel results in the field. No tests were run for air entrainment at that time but tests on the cement at a later date showed that the grinding compound was about 0.05 per cent. With one bag replacement the slight loss in compressive strength of only 4 per cent and the slight increase of 1 per cent in the flexural strength indicates that the air entrainment was low.

The following reports from records of the New York State Department of Public Works relative to the Wellsville-Bolivar job laid several years ago in Allegany County in which twenty different portland cements were used with and without grinding aid. On that road the standard portland cement averaged 149.1 lb. per cu. ft. The average of the vinsol resin concrete was 145.5 lb. per cu. ft. The average weight of the portland cement with natural cement designated as No. 1 in the Timms' report was 148.3 lb. per cu. ft.

The average decrease in weight below the standard portland cement was 1.1 lb. per cu. ft. with the natural cement No. 1 as against 3.9 lb. for the vinsol resin concrete.

In New York State Laboratory freezing-and-thawing tests, the average life of the normal highway concrete mix of vinsol resin portland cement was 119.3 cycles. The average life of the local portland cements meeting New York State Specifications for portland cement with the blend of natural cement No. 1 was 156 cycles, indicating again that high air entrainment is not necessary if good quality portland and natural cements are used.

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[12] Presented at the March 18, 1940 meeting of Committee C-1 in Washington, D. C. not published.

A recent survey has been completed by the State Department of Public Works on the concrete laid on the Storm King highway in Orange County in 1939–1940. The average weight of concrete in the case of one 94 lb. bag replacement was 151.56 lb. The average weight with the vinsol resin portland cement was 146.25 lb. per cu. ft.

The losses in freezing and thawing in calcium chloride solution at the age of ten months varied from 100 per cent loss at 25 cycles for the poorer portland cements to 100 per cent loss at 100 cycles for the best of the portland cements.

With all of these portland cements to which either Vinsol Resin or natural cement had been added, the loss at 100 cycles of freezing and thawing in the calcium chloride solution varied from 1.2 per cent to 14.8 per cent with an average of approximately 5 per cent.

The results of the survey show that all natural and vinsol resin sections are in excellent condition with little or no scale, again proving that high air entrainment is not necessary when good cements are used.

The above are only a few of the hundreds of tests which prove that high air entrainment is not necessary and that with good materials and low air entrainment excellent resistance to freezing and thawing may be obtained without high loss in stengths.

In the northeastern territory no cements can be relied upon in all cases to give the desired results unless a small amount of waterproofing is added, and a study of this subject should be carried much further in order to determine how much waterproofing is required with the different quality cements.

All portland cements are not equal and all natural cements are not equal. It is a fallacy to treat them as such. All grinding aids and waterproofing aids are not equally effective.

In this connection reference is made to a question raised by F. H. Jackson in his article on the "Durability of Concrete in Service" appearing in the *Journal*, American Concrete Institute, October, 1946 in which he found on an inspection of concrete structures that in concrete built previous to 1930, 67 per cent were free from serious deterioration while on the structures built subsequent to 1930 only 27 per cent could be so classified.

Mr. Jackson also reported results from a survey by Glen Paxson, Bridge Engineer of the Oregon State Highway Department.

Mr. Paxson reported 78 per cent good on the earlier structures with only 37 per cent good on the later structures.

Mr. Jackson raised the question:

"In view of these facts is it not surprising that engineers should wonder about the cement?"

I believe the quality of the cementing material is important and that concrete consumers should not be encouraged to rely on high air entrainment for their ultimate results. It has not yet been proved, when good cements are used, that because a little waterproofing or air entrainment is of value that a lot is better.

MESSRS. TIMMS, GRIEB, AND WERNER *authors' closure, by letter).*—The extensive discussion of the paper has added greatly to a better understanding of the use of blends of natural and portland cement.

There appears to be a difference of opinion among those who discussed the paper as to the reasons given as an explanation of the behavior of the blends. The authors believe the data given in the paper clearly show that the improved resistance to freezing and thawing of the blends is a function of the air entrained in the concrete. However, attention again is called to the fact that the tests were conducted on test specimens laboratory fabricated and cured.

SURFACE AREAS OF HIGH-CALCIUM QUICKLIMES AND HYDRATES*

By H. R. Staley[1] and S. H. Greenfeld[1]

Synopsis

Using surface area as a means of evaluation, the wet slaking characteristics of quicklimes made from a high-calcium stone were studied. Quicklimes produced under two calcining conditions, representative of commercial rotary (S series) and vertical (H series) kiln products, and in the presence of sodium chloride, were slaked at temperatures ranging from 5 to 113 C. (41 to 235 F.) and with water from one to twenty times the minimum required for complete hydration. Hydration in the presence of ethanol and sucrose was also studied.

When slaked with excess water, lime hydrates produced from "hard-burned" quicklimes had higher surface areas than those made from "soft-burned" quicklimes. The surface area of the lime hydrates made from either quicklime increased with slight excess water of slaking, but with increasing amounts of excess water the surface areas dropped rapidly to constant values. This latter figure for the H series was about twice as large as that obtained for a hydrate produced with the minimum water of hydration; for the S series it was somewhat lower than that obtained by "dry" hydration.

When the quicklimes were slaked with eight times the minimum water requirement, in both series the surface areas passed through a minimum as the temperature of slaking increased from 20 to 100 C.

Quicklimes made in the presence of NaCl under relatively low-temperature conditions were similar to "hard-burned" quicklimes, but gave a low surface area upon wet hydration.

Vapor-phase hydration was extremely slow and produced a hydrate of low surface area.

The use of both ethanol and sucrose solutions produced high surface area wet hydrates, but had no effect on the dry-hydrating characteristics.

In spite of the long history of the production and use of lime, there are many imponderable factors which have never been precisely defined. Presumably, because of its ready availability and cheapness, many producers and consumers of this widely used chemical or structural material have been satisfied to manufacture and use lime without realizing, or without making an attempt to evaluate quantitatively, its full potentialities or characteristics. As a result, the impression that "lime is lime" is a somewhat prevalent and, in general, an erroneous belief.

It has been shown by Azbe (**1**)[2] that the density of a quicklime may vary from 1.46 to 3.00 according to the conditions imposed in calcination. It is known that the "soft-burned" product is much more reactive with water than the "hard-burned," and it follows that the "hard-burning" causes a reduction in surface activity. This lower surface activity may be due merely to a decrease in surface, either external or internal, or due to some modification in surface structure which decreases the affinity of the surface for water. It is also known that the "soft-burned" material retains approximately its original bulk volume, while the bulk volume of the "hard-burned" material is much lower than that of the stone.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

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[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 964.

The difference in activity of the quicklimes influences markedly the properties of the hydrates. Hydrates produced from "soft-burned" quicklimes are much finer than those produced from "hard-burned" quicklimes. However, if suitable methods of hydration are used, a finer product may be obtained from the latter.

In order to get a quantitative idea of the phenomena involved in the production of lime hydrates, the burning and wet-slaking characteristics of a relatively pure high-calcium limestone were studied. Wet slaking was chosen because no suitable, reproducible, dry-slaking process had been developed at the time. In order to get improved products, several slaking aids, such as ethanol and sugar, were also investigated. The criterion of quality used is the surface area of the sample, as obtained by the low-temperature nitrogen adsorption method, calculated according to the Brunauer, Emmett, and Teller theory (**3, 4**). A distribution function which is indicative of the pore-size variation was also calculated with the aid of this theory.

EXPERIMENTAL TECHNIQUE

Materials:

For the surface-area measurements a medical grade of helium was used to determine the dead space in the sample bulb. The helium was taken into the gas reservoir through a liquid nitrogen trap filled with activated charcoal to remove any impurities which might be adsorbable. Further check of the reliability of the dead-space value was its determination at two different pressures.

The nitrogen was a water-pumped grade. In the early runs it was fed through a dry-ice bath, but as the absence of the bath produced no change in the results, it was not used during these runs.

The high-calcium limestone used to produce the quicklimes and hydrates used in this study had the following average analysis:

$CaCO_3$	98.5 per cent
$MgCO_3$	0.5 per cent
SiO_2	0.4 per cent
R_2O_3	0.4 per cent

Apparatus and Procedure:

The stone was burned in a muffle-type glo-bar furnace designed specifically to maintain a uniform temperature throughout a large portion of its interior. The temperature was measured with a Leeds & Northrup optical pyrometer through a peep-hole in the door of the furnace. The furnace was raised to proper temperature before the stone was inserted for calcination. As the ½ to ¾-in. stones were put on the floor of the furnace there was a temperature drop of about 50 F. (28 C.), but as the quantity of stone was small, the temperature soon was at the desired level again. An error of several minutes may thus be assumed in the time of burning.

The hydration process was carried out in glass beakers suspended in a water bath maintained at a temperature somewhat lower than the required temperature of hydration, in order to maintain the proper temperature within the beaker. The suspension of solid in liquid was stirred constantly with a thermometer and was kept within ± 3 C. of the temperature desired. After hydration was complete, which was indicated by no further increase in temperature, the material was cooled to room temperature and the suspension was filtered, washed with alcohol, and dried at 110 C. The alcohol wash minimized the drying time and decreased the tendency for any cementation to occur in the oven.[3]

Complete hydration was checked on several of the samples by re-wetting at about 60 C. for 30 min. and noting no gain in weight upon drying.

The surface areas and isotherms were obtained in the apparatus shown in Fig. 1, which is similar to that used by Brunauer, Emmett, and Teller (**3**). The surface areas and pore sizes were evaluated by means of the Brunauer, Emmett, and Teller theory.

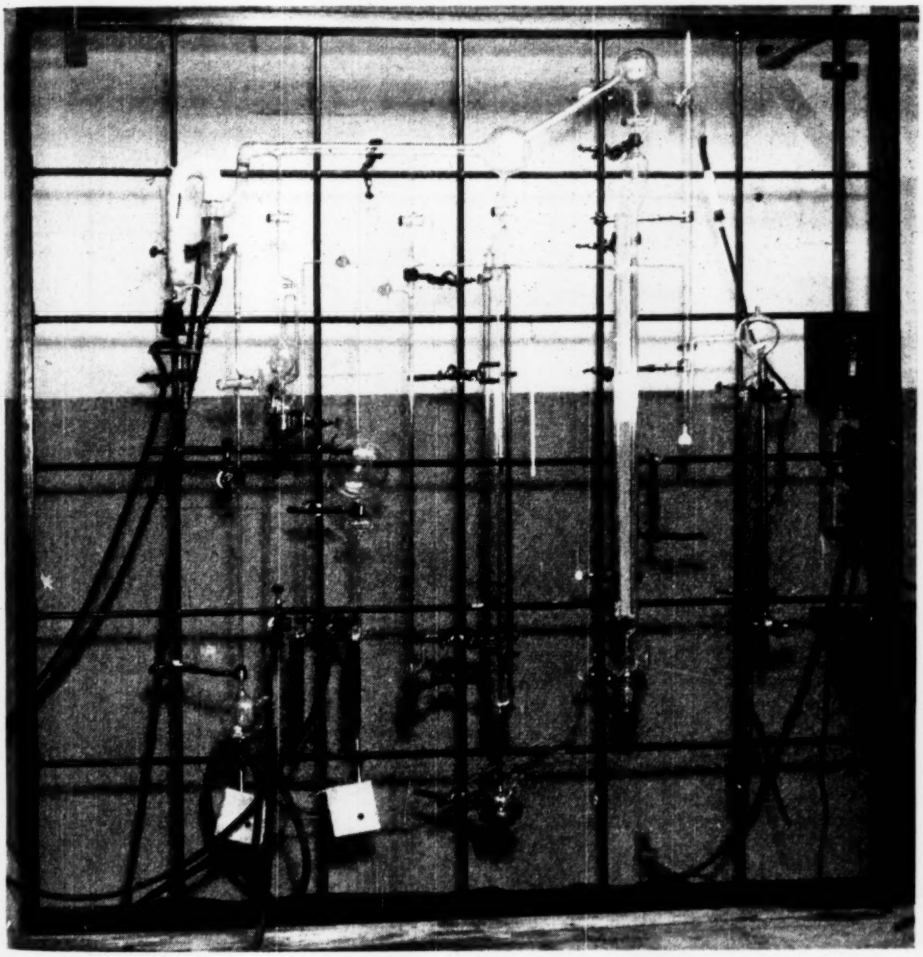

FIG. 1.—Surface Area Apparatus.

Brunauer and his co-workers, in two papers (**3, 4**), developed a theory of multimolecular adsorption based on the kinetic theory. The derivation of the following equation may also be found in Brunauer's book (**2**).

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[3] It was found that even though the alcohol wash changed the appearance of the product, made it light and fluffy rather than caked, it did not change the surface area.

$$V = \left(\frac{V_m\,cx}{1 - x}\right)\left(\frac{1 - (n + 1)x^n + nx^{n+1}}{1 + (c - 1)x - cx^{n+1}}\right) \quad (1)$$

where:

V = volume of gas adsorbed at any x,
x = P/P_o, relative pressure,
P = pressure,
P_o = vapor pressure of adsorbate at the temperature of the sample,
c = a constant related to the average heat of adsorption,

$$c \cong e^{(E_1 - E_2)/RT}$$

E_1 = heat of adsorption,
E_2 = heat of condensation of adsorbate,
R = gas constant,
T = adsorbent temperature,
V_m = gas adsorbed at the completion of the monolayer, and
n = maximum number of layers which can be adsorbed on each wall of the capillaries.

If the material is relatively nonporous, n may be assumed equal to infinity and the equation, upon rearrangement, becomes:

$$\frac{P}{V(P_o - P)} = \frac{1}{V_m\,c} + \frac{(c - 1)P}{V_m\,c\;P_o} \quad \ldots (2)$$

In the case of quicklimes and lime hydrates, the smallest pores seem to be greater than 20 Angstrom units and Eq. 2 can be applied below $P/P_o = 0.25$, where these small pores become filled and begin to limit adsorption. However, since the heat of adsorption is much greater on the more active portions of the surface (which are occupied by the first portions of gas adsorbed), and the constant c applies only over the range where the heat of adsorption is relatively constant, neither Eq. 1 nor Eq. 2 holds below $P/P_o = 0.05$.

Keeping the above limitations in mind, one can determine the constants V_m and c from Eq. 2 from two values of V and P in the range of P/P_o from 0.05 to 0.25. In practice, at least three values are recorded in this range (as a check). The term $\dfrac{P}{V(P_o - P)}$ is plotted against P/P_o and the constants are determined from the slope, $\dfrac{c-1}{V_m c}$, and the intercept, $\dfrac{1}{V_m c}$. Using these constants in Eq. 1, isotherms can be calculated for various values of n. When the experimental isotherm is compared with these calculated isotherms, the points at which the capillaries of various sizes become filled can be seen; the smaller capillaries are filled first, and as each larger size capillary is filled, it no longer limits adsorption, the limiting capillary then being the next larger one. Because of the peculiar shape of the isotherms this method is not quantitative for the determination of pore-size distributions, but it is an excellent method for qualitative comparisons of the smaller capillary size ranges.

For the purposes of this work, it is assumed that adsorption takes place equally on all walls of the capillaries, permitting $2n$ layers in any capillary. The diameter of the nitrogen molecule is considered to be 3.15×10^{-8} cm. $(10^{-8}$ cm. = 1 Angstrom unit). Each calculated isotherm corresponds to a material having pores (2) (3.15) (n) = 6.30 n Angstroms in diameter. As the material under consideration actually has pores of many different sizes, the experimental isotherm corresponds to none of the calculated isotherms. However, as it is the smallest unfilled pores which limit adsorption at any particular relative pressure, (P/P_o), the experimental isotherm will follow a calculated isotherm while its pores of that particular size are being filled. Hence, as the smaller pores become filled, the experimental isotherms deviate upward to intersect calculated isotherms for increasing values of *n*. Figures 4, 5, and 6 show the superposition of the experimental isotherms of several samples on their *n* isotherms.

TABLE I.—QUICKLIMES.

Designation	Treatment,[a] Time and Temperature	Surface Area, sq. m. per g.	Pore Size, Angstrom Units
Stone	None (−9 mesh material)	0.57	35 up
HQ	15 hr. at 2400 F.	0.44	45 up
H_1Q	15 hr. at 2250 F.	0.45	
SQ	2 hr. at 2400 F.	1.18	20, Majority 30 up
S_1Q	4 hr. at 2200 F.	1.30	
S-NaCl	3 hr. at 1950 F. with 0.5 per cent NaCl	0.67	50 up
S_1-NaCl	2 hr. at 2300 F. with 0.5 per cent NaCl	0.55	38 up

[a] 1/2 to 3/4-in. stone was used and the quicklime was crushed to −9 mesh for purposes of handling.

TABLE II.—WET HYDRATES.

Designation[a]	Surface Area, sq. m. per g.	Designation[a]	Surface Area, sq. m. per g.
H-20-8x	30.2	S-5-8x	6.7
H-30-8x	26.1	S-20-8x	24.4
H-40-8x	27.3	S-40-8x	19.2
H-60-8x	30.4	S-60-8x	16.2
H-80-8x	30.8	S-80-8x	17.8
H-100-8x	32.2	S-100-8x	22.5
H-60-4x	31.8	S-60-4x	21.5
H-60-20x	29.2	S-60-14x	17.5
		S-60-20x	17.4
		S-60-D	22.1

[a] The H series of hydrates was made from quicklime HQ and the S series, from SQ. The numbers following the capital letters refer to the temperatures (deg. Cent.) at which the hydration process took place and the numbers immediately preceding the "x" refer to the amount of water used in terms of the minimum requirement for complete hydration. Theoretically, 32.1 g. H_2O per 100 g. CaO is sufficient, but actually to produce a dry, completely hydrated product 52 g. H_2O per 100 g. CaO were required.

RESULTS

The surface areas and estimated pore sizes of the limestone and quicklimes produced by the various calcining conditions are given in Table I.

Table II gives the surface areas of the lime hydrates produced by varying the amount of excess water used in slaking and the temperature of slaking. The effect on surface area of the inclusion of ethanol and sucrose in the slaking water is shown in Table III.

Other hydrates were produced, as given below, to determine the effects upon surface area of hydrating quicklime with water vapor and hydrating quicklime to a dry end product in the presence of sucrose. The effect of the presence of salt (NaCl) during calcination upon the surface area of two hydrates was also determined.

TABLE III.—MODIFIED HYDRATES.

Designation[a]	Surface Area, sq. m. per g.	Designation[b]	Surface Area, sq. m. per g.
HA-10-50	33.3	HS-2	41.0
HA-50-35	45.6	HS-1/2	37.0[c]
SA-10-50	28.3	SS-1/2	44.3
SA-25-80	36.0	SS-2	46.3
SA-50	38.0	SS-5	28.9
SA-25D (Dry Hydrated)	21.4		

[a] SQ and HQ hydrated with eight times the minimum amount of ethanol-water solutions of concentration designated by the first number, reaching a temperature designated by the second number.

[b] SQ and HQ hydrated at 60 C. with eight times the minimum amount of sucrose solution of percentage composition designated by the number.

[c] At 100 C.

Special Hydrates:

Hydrate	Treatment	Surface Area
SD-30	Quicklime SQ hydrated in a desiccator with saturated air at 30 C.	10.4 sq. m. per g.
SD-113	Quicklime SQ hydrated at 113 C. with steam	8.1 sq. m. per g.

Modified Hydrates:

Quicklimes SQ and HQ hydrated with 1.25 times the minimum water requirement with the concentration of sucrose solution designated by the first number, reaching a temperature designated by the second number:

	Surface Area, sq. m. per g.
Hydrate HS-2-130	12.4
Hydrate SS-½-100	21.4

The quicklimes S-NaCl and S_1-NaCl

made in the presence of sodium chloride hydrated with eight times the minimum required water at a temperature of 60 C.:

	Surface Area, sq. m. per g.
Hydrate SN-60	17.6
Hydrate S_1N-60	14.6

DISCUSSION OF RESULTS

Quicklimes:

In the dissociation of a limestone, it is logical to assume that if the conditions of calcination are such that a minimum of heating occurs after the carbon dioxide has been driven off, and little or no recarbonation occurs, the quicklime should have a maximum surface area and should exhibit much smaller minimum size pores than the original limestone from which it was made. While such conditions of calcination may or may not be an objective, depending upon the type of product being produced, it is recognized that these conditions are usually impossible of attainment for calcination of commercial products according to orthodox methods; therefore, for this study, conditions of calcination were chosen which were more closely analogous to commercial practice.

Considering the SQ quicklimes, it is seen that the surface area has been increased and the minimum pore size much reduced by the calcination. It is indicated, in the difference between SQ and S_1Q, that the temperature is much more significant than time of burning, if surface area may be taken as a criterion.[4]

The "hard-burned" products HQ and H_1Q have surface areas even less than the original stone and a minimum pore size somewhat larger than in the stone. The shrinkage, or possible fusion due to impurities, that occurs under these conditions of calcination actually closes the small pores, thus accounting for the reduction of surface area. From a comparison of the surface areas of the "hard-burned" and "soft-burned" quicklimes, it is not difficult to see why one is more reactive with water than the other. It is not evident what portion of this decrease in reactivity of the "hard-burned" material is due to an actual decrease in surface area, and what portion is due to decreased activity of the surface, but it has been shown that many materials possess spots or local areas that are much more active than other portions and that these active areas can be destroyed by heat in some instances. It is not too illogical to assume that the same phenomenon can occur in the case of limes.

Previous work done by Sawai and Yoshioka (**5**) indicated that lime calcined in the presence of salt is difficult to hydrate. The two samples, S-NaCl and S_1-NaCl, calcined in the presence of 0.5 per cent NaCl under conditions of temperature analogous to "soft-burning" exhibit surface areas similar to "hard-burned" products. It is indicated in this case also that temperature is a more significant factor than time in the plugging of pores. The low surface areas account, at least partially, for the slow rate of slaking but it is believed that there are other surface phenomena involved which help to reduce the reactivity. These quicklimes were much harder than regular "soft-burned" quicklimes and had a yellow tint. The pore-size functions show that the small pores have disappeared, as in the case of the "hard-burned" quicklime. Sodium chloride has apparently acted as a fluxing material, increasing the mobility of the calcium and oxygen atoms or ions sufficiently to permit them to pack more tightly and more rapidly than in its absence.

---

[4] Surface area measurements of closely sized fractions of material of this sort indicate that the walls of the pores contribute a major portion of the area. Within the accuracy of measurement, the surface area is independent of particle size above the 200-mesh size. The precision of measurement of the surface is ± 1 per cent while the accuracy of the theory is within 5 per cent. The surface area determined is in distinct contrast to that determined by the Wagner Turbidimeter, for example, which gives only a *relative* figure which is dependent upon the effective diameter of the particles in the medium in which the material is suspended.

However, the quicklimes calcined in the presence of salt are not entirely like "hard-burned" materials, as is evidenced by their wet-slaking characteristics; these are much more like the "soft-burned" quicklime. (Since a dry-slaking process which gives reproducible results had not been perfected at the writing of this report, the dry-slaking characteristics of the material are not reported, but the dry-hydrated materials are much different from the wet hydrates discussed here.)

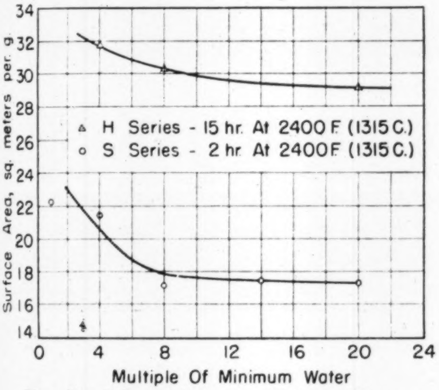

FIG. 2.—Effect of Excess Slaking Water on Surface Area of Hydrates.

Material—High-Calcium Quicklime. Slaking Temperature—60 C.

Wet Hydrates:

Although in commercial practice and in the laboratory it has been found that hydrates made by dry hydration from "soft-burned" quicklimes have higher surface areas than those made from "hard-burned" quicklimes, it is readily seen from the data that the reverse is the case when wet hydration is used.[5] The hydrates made from the "hard-burned" quicklimes by wet hydration had surface areas approximately double those of commercial dry hydrates; those made from the "soft-burned" quicklimes by wet hydration had somewhat lower surface areas than commercial dry hydrates. The hydrates S-100 and S-60-D, with surface areas of 22.5 and 22.1 sq. m. per gram, respectively, are indicative of the surface area which would be obtained with ordinary dry hydration. As the water in excess of that required for complete hydration is increased, the surface area drops off sharply for the S series and not quite so sharply for the H series (Fig. 2). It was rather unfortunate that 60 C. was chosen as the temperature for the excess-water hydrations, for later it was found that the surface areas of the "soft-burned" samples go through a minimum in that vicinity. However, it was easy to hold the temperature at 60 C., making it convenient during the hydration process.

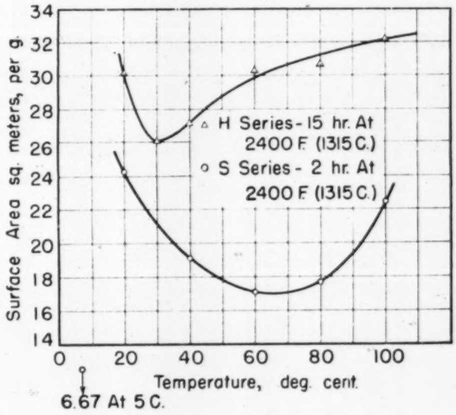

FIG. 3.—Temperature Effect for Wet Slaking.

Material—High-Calcium Quicklime, Eight Times Minimum Water

Figure 6 shows the effect of temperature on wet hydration, using eight times the minimum water requirement (see footnote under Table II). In both series the surface area goes through a minimum. In the H series it is a sharp minimum while in the S series it is not so abrupt. Although not shown, the curves must go through a maximum also, somewhat below 20 C., for sample S-5 has a surface area of 6.67 sq. m. per gram and, even though completely hydrated, looks similar to the quicklime from which it was made.

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[5] Hydrates from commercial rotary kiln quicklimes have surface areas of approximately 20 sq. m. per gram while hydrates from vertical kiln quicklimes are seldom above 16 sq. m. per gram.

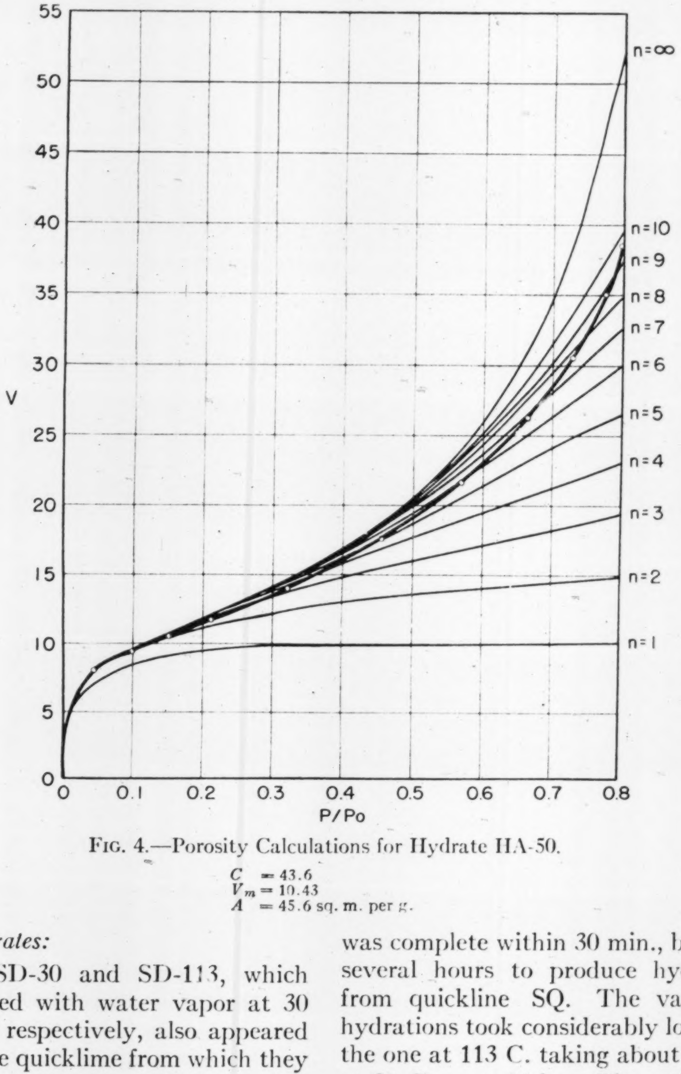

FIG. 4.—Porosity Calculations for Hydrate HA-50.

$C = 43.6$
$V_m = 10.43$
$A = 45.6$ sq. m. per g.

Special Hydrates:

Samples SD-30 and SD-113, which were hydrated with water vapor at 30 and 113 C., respectively, also appeared much like the quicklime from which they were made. However, their surface areas were increased to 10.4 and 8.1 sq. m. per gram, a considerable increase over that of the quicklime, which was 1.18 sq. m. per gram. The low surface area and "dead" appearance of these last three hydrates are true indications of their poor quality. Vapor-phase hydration has never proved successful in commercial or laboratory practice.

In all cases above 20 C., wet slaking was complete within 30 min., but it took several hours to produce hydrate S-5 from quickline SQ. The vapor-phase hydrations took considerably longer also, the one at 113 C. taking about 8 hr. and at 30 C., over 24 hr. These times were rather long compared to the two or three minutes required to hydrate a "softrned" quicklime under normal slaking nditions. The unusually long time as the result of keeping the temperature own to its specified level in the case of 5 and SD-30. The slow hydration of mple SD-113 probably indicates that e hydration reaction is ionic in charac- er and it is difficult for ionized water to xist on the solid surface at 113 C. The gher the temperature of hydration, the ore rapidly the process took place. owever, Fig. 3 shows that the surface ea goes through a minimum as the mperature goes up. Although no data are available, judging from the curve and from the known dry-slaking characteristics, it might be predicted that for the S series the minimum would shift to the left as excess water was increased and in the H series it would shift to the right. In both cases the curves would be displaced somewhat upwards, also, until about 1.5 minimum water is reached; and then they would be displaced downward rapidly.

In an attempt to obtain hydrates of higher surface area, it was decided that a material which would decrease the solu-

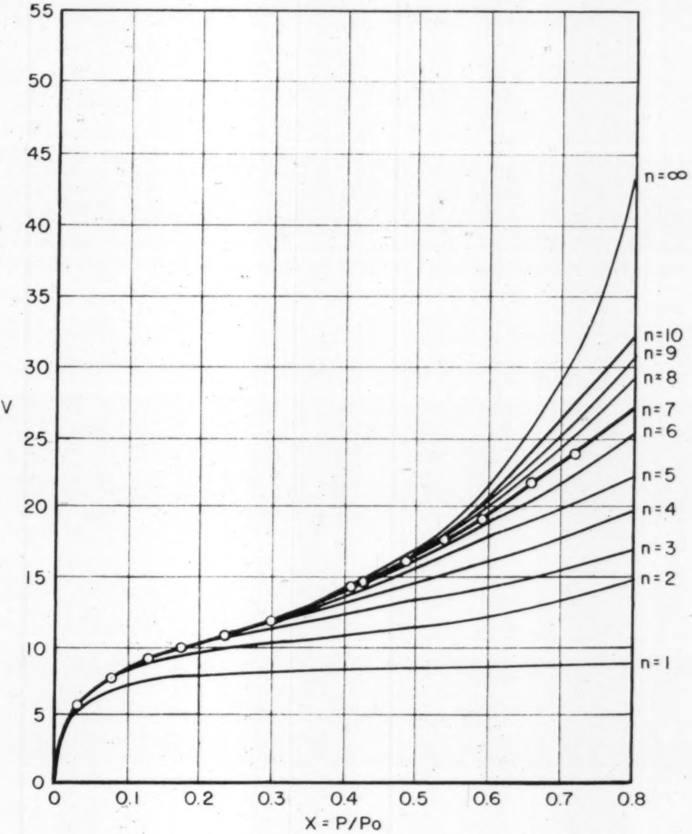

FIG. 5.—Porosity Calculations for Hydrate SA-50.

$C = 64.9$
$V_m = 8.71$
$A = 38.0$ sq. m. per g.

bility of the lime should increase the surface area by minimizing cementation and pore plugging during drying. The series of runs made with alcohol was the result. For both the H and S series it can be seen that the surface area increases with increasing percentage of alcohol. No definite conclusions can be reached, except that the surface area increases, for no attempt was made to hold the temperature constant and the total liquid to solid ratio was permitted to increase with the percentage of alcohol in order to keep the water-solids ratio constant. Figures 4 and 5 indicate that for the H series, the pores in the hydrates are of varying sizes while in the S series the pores are practically of uniform size ($n = 7$ for hydrate SA-50). The phenomenon described above indicates that where particle growth and pore-plugging cannot take place, as is the case when material is constantly dissolving and reprecipitating, the pores of the quicklime will determine the pores of the hydrate. The "soft-burned" quicklime had fairly uniform pores of small diameter (from the escaping CO_2) while the "hard-burned" quicklime had larger pores of varying diameter.

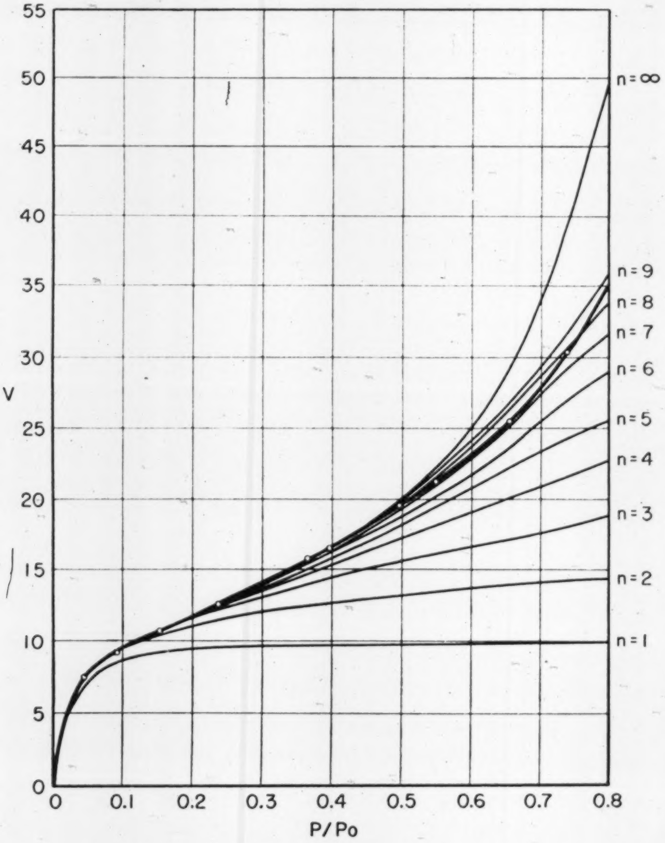

FIG. 6.—Porosity Calculations for Hydrate SS-½.

$C = 61.7$
$V_m = 10.13$
$A = 44.3$ sq. m. per g.

To check the above contention it was decided to make some runs using a solubilizing agent, such as sucrose. It was realized that the size of the molecule would hinder particle growth and thus produce a higher area, but the solubilizing should produce varying pore sizes in both series of hydrates. Figure 6, of hydrate SS-$\frac{1}{2}$, indicates this to be the case.

Dry hydration in the presence of alcohol or sugar seems to have little effect, for the surface areas are of the same magnitude as the quicklimes dry hydrated with water alone.

Figure 7 shows the relation between the volume adsorbed and the pore-size "limiting" adsorption. It is seen that these distributions, which are indicative of the pore-size distributions in the samples, are random in nature. The only noticeable trend is that as the surface area goes up the slope of the curve becomes greater.

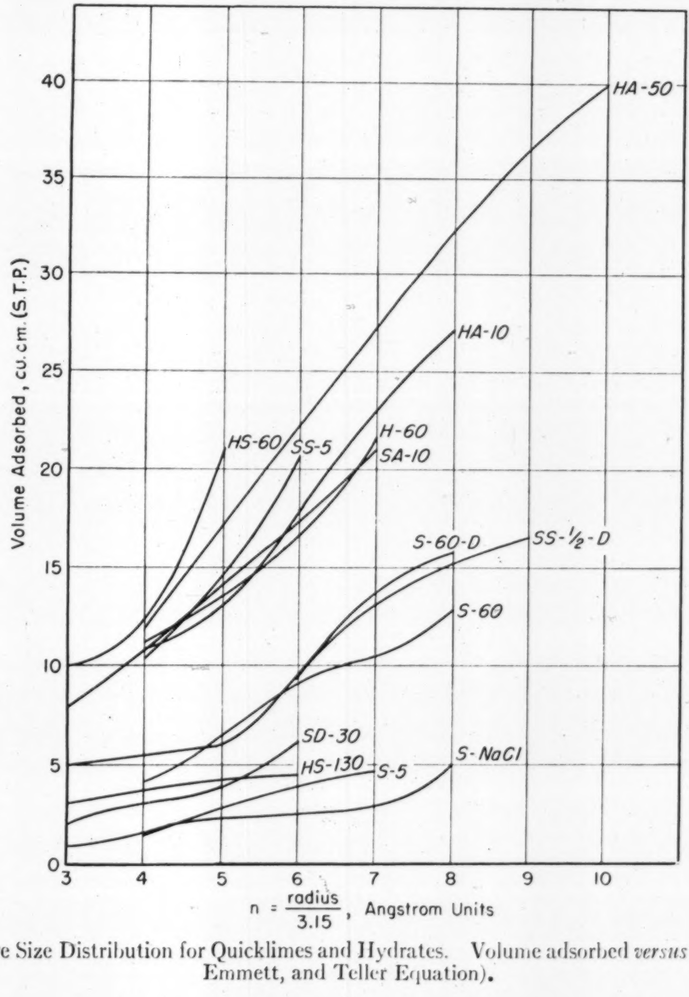

FIG. 7.—Pore Size Distribution for Quicklimes and Hydrates. Volume adsorbed *versus* n (Brunauer, Emmett, and Teller Equation).

CONCLUSIONS

It was not possible to include all of the possible calcining temperatures and conditions of hydration in the scope of this investigation but sufficient data were obtained to indicate significant trends as to the effect of certain variables on the properties of the resulting material. It is apparent that the modification of surface area, due to changes in calcining or hydrating conditions, is largely the result of the modification of pore size and structure since a large percentage of the total surface area, as measured, represents internal surface.

A more comprehensive program of research is under way in which pore sizes and distributions as well as other adsorption characteristics are being studied. The results of these studies will be reported when available.

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DISCUSSION

MR. J. A. MURRAY[1].—This paper is a valuable contribution in showing the applicability of a relatively new research tool to an old industry and pointing the way to a better understanding and possible solution of some of the problems facing the industry. Interpretation of the results is somewhat difficult as the authors have used an isothermal slaking procedure whereas in practice lime is slaked either at 100 C. or else under conditions which are substantially adiabatic.

This is important in considering the data in Table II and Fig. 3. The majority of tests were made with eight times as much water as was required for complete hydration. It is not clear whether this refers to the 32.1 g. H_2O per 100 g. CaO or to the 52 g. H_2O per 100 g. CaO referred to in the footnote of Table II. If the former, the factor 8x represents 257 g. H_2O per 100 g. CaO; if the latter, the factor represents 416 g. H_2O per 100 g. CaO.

It may readily be calculated that the amount of water required to slake 100 g. of CaO and raise the resulting mixture just to 100 C. but with no steam evolution is about 300 g. if the initial reactants were at 20 C. Hence, if the factor 8x refers to 257 g. of water, every one of the tests so marked, in Table II, required cooling. On the other hand, if the factor 8x refers to 416 g. of water, this is very nearly the amount required to give a resultant temperature under adiabatic conditions of 80 C. Hence the tests at 100 C. would require supplemental heat while those at 60 C. and below would require cooling. It is conceivable that the minimum shown in Fig. 3 may be caused by this variation in conditions and might not be evident if the lime were slaked under conditions more similar to those used in practice.

The data in Tables II and III are reported to the nearest 0.1 sq. in. per gram. Presumably, this refers to the average of replicate samples of each hydrate. It would be of interest to know the reproducibility that could be obtained by replicate hydrations of one quicklime; and by replicate burning and hydration of one limestone.

Some data are given on "Special Hydrates," one of which was hydrated with steam at 113 C. This temperature may be misleading. Hydrating dolomitic quicklime in a steam atmosphere at 15 psi. pressure (122 C.) with a thermocouple inserted in the mass of lime will result in internal temperatures above 300 C.

MR. HOWARD R. STALEY (*author's closure*).—As stated in the foot ote to Table II, the minimum (not the theoretical) required slaking water is 52 g. per 100 g. of CaO. The number preceding the "x" in the table refers to multiples of this minimum. Heating or cooling was necessary in almost all of the hydrations in order to maintain the temperature at ± 3 C. of the desired temperature.

Slaking under adiabatic conditions would probably result in lower surface areas than were obtained in these tests. Earlier tests indicate this to be the case.

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[1] Research Director, Warner Co., Devault, Pa.

Duplicate burning and slaking of carefully selected stone of one sampling yielded surface areas within ± 3 per cent. It is true that if care were not exercised in the selection of the stone and in control of burning and slaking, the percentage variation in surface area was greater than ± 3 per cent.

All of the quicklimes were crushed to minus 8 mesh before slaking, and it is believed that the temperatures of slaking reported are close to the actual values. While some differential in temperature can be expected in the mass, the lumps were small enough not to have a large difference.

The quicklime hydrated in steam was hydrated in an oven, maintained at 113 C., in a saturated atmosphere, produced by the addition of steam.

METHODS FOR THE DETERMINATION OF SOFT PIECES IN AGGREGATE*[1]

By D. O. Woolf[2]

In 1941, Subcommittee IX of the Society's Committee C-9 on Concrete and Concrete Aggregates requested that an investigation be made of methods of testing coarse aggregates to determine the content of soft pieces. It was hoped that the results of this investigation would direct attention toward the development of a new method of testing aggregates for soft pieces, or would identify existing methods suitable for this purpose.

This paper describes the methods employed and gives typical test values obtained. With each method of test are presented the conclusions obtained from the test data: whether the method appears to be useful, and the difficulties encountered in the performance of the test. Efforts have been made to develop a method suitable for use in the field. The detection of the amount of soft pieces in aggregates is properly a duty of the inspector in the field; consequently, the program of the investigation was planned with consideration of field use as a primary requisite. Any method which the inspector can use can also be performed in the laboratory. Some of the methods employed apparatus suitable for use in the laboratory only. In most of these cases, however, plans were prepared for the construction of equipment sufficiently portable to permit its installation at aggregate plants.

Classification of Materials

Prior to this investigation, information was solicited on materials which the various highway authorities classify as soft pieces. The State highway departments were requested to submit samples of the soft pieces encountered in their own states, and to furnish information relating to their particular conditions. The following information was requested:

1. The names of the different materials which may be classified as soft pieces.
2. The kinds which are found generally throughout the state or in large areas of the state.
3. The amount of soft pieces permitted in specifications for surface courses.
4. Method of test or identification used by laboratory operators or field inspectors (with descriptions if such are available).
5. The materials occurring naturally which are considered to be objectionable for surface courses but which are not included in the class of soft pieces.

Based on the information received, an alphabetical list of materials classed as "soft pieces" is given in Table I. The table also shows the number of states which classify the material as soft, and also the number of states which report the material to be widely distributed.

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*Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] A Report to Subcommittee IX on Specifications and Methods of Tests of Aggregates of A.S.T.M. Committee C-9 on Concrete and Concrete Aggregates.

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There are seeming incongruities in Table I. For example, granite is placed in the classification of soft pieces by two states, although to most persons the word "granite" conveys the impression of a hard and enduring stone and other apparent inconsistencies might be mentioned. However, some interpretation of these classifications is probably permissible. There is little doubt that the inclusion of granite in a classification of soft pieces is meant to refer to material of a granitic nature which has weathered to such an extent that the component crystals are soft or poorly bonded to each other. Similar interpretations can be made of a number of the materials included in the table, and, in general, most of the materials mentioned can be placed in one or more of the following types:

1. soft,
2. unsound,
3. light weight,
4. brittle,
5. friable, and
6. highly absorbent.

TABLE I.—LIST OF MATERIALS CLASSED AS "SOFT PIECES" AND NUMBER OF STATES GIVING THIS CLASSIFICATION OR HAVING MATERIAL WIDELY DISTRIBUTED.

Material	Number of States		Material	Number of States	
	Classifying as soft	Wide distribution		Classifying as soft	Wide distribution
Basalt, disintegrated	1	1	Limestone, soft	3	1
Brownstone	1	1	Limestone, some varieties	1	1
Caliche, hard	1	0	Limestone, weathered	4	3
Chert	2	1	Limonite	3	2
Chert, chalky	1	0	Marble	1	1
Clay balls	2	1	Ochre	3	2
Clay, iron bearing	1	0	Quartz, sugar	1	1
Coal	2	1	Quartz, weathered	2	2
Concretion, calcareous	1	1	Rock, disintegrated	2	1
Conglomerate	1	0	Rock, weathered	2	2
Dolomite, weathered and porous	1	1	Rottenstone	1	1
Earth, diatomaceous	1	1	Sandstone	9	9
Feldspar	1	1	Sandstone, argillaceous	1	1
Felsite	1	0	Sandstone, friable	1	1
Floaters	1	1	Sandstone, friable arkosic	1	0
Gneiss	1	0	Sandstone, soft	5	4
Gneiss, micaceous	1	1	Schist, some	5	3
Gneiss, weathered	1	1	Schist, some	1	1
Granite	2	1	Schist, micaceous	1	0
Granite, decomposed	1	1	Schist, talc	1	0
Granite, disintegrated	4	3	Schist, weathered	2	2
Granite, micaceous	1	1	Scoria	1	1
Granite, weathered	3	3	Scoria, certain grades of	1	1
Gravel, cemented	2	1	Shale	11	7
Gravel, magnesia	1	1	Shale, clay	1	1
Iron clay balls	1	1	Shale, diatomaceous	1	1
Iron oxide	2	1	Shale, disintegrated	1	1
Limestone, argillaceous	2	1	Shell	2	1
Limestone, porous	1	1	Soapstone	1	0
Limestone, shelly	1	0	Volcanic rock, coarse	1	1

As indicated above, some materials may be both soft and friable, or soft and brittle, or different samples of the same material may be placed properly in separate groups. Consequently, without reference to actual samples, a proper grouping of materials by name alone is probably not feasible. Certain materials, such as brownstone, micaceous granite, and limonite mentioned in Table I, are not identified definitely by the names given although the use of these terms may be justified on the basis of limited geographical usage. The desirability of applying more widely used, scriptive terms to describe materials of inite physical characteristics, is evi- nt, as is the necessity for the establish- ent and use of a systematized glossary terms relating to the materials under nsideration.

Table II presents a list of materials assed as deleterious but not considered s soft pieces by the reporting authority. t is interesting to note that in this list f 37 entries, 17 are placed in the cate- ory of soft pieces by other states. This ustrates one of the difficulties encoun- ered in attempting to coordinate equirements or tests for soft pieces in aggregate, for unless there can be some agreement as to what constitutes a soft piece, work along the proposed line may not be of suitable value.

BLE II.—LIST OF MATERIALS CLASSED AS DELETERIOUS OTHER THAN SOFT PIECES.

aline reactive
phibolite
ert
ert, opaline
ert, unsound
ay lumps
al
ated material
olomite, some
int
assy rock
eiss, soft
anite
anite, soft
anite, some
avel, a quartz
ydrophilic rock
mestone, argillaceous
mestone, siliceous
Mica
Mud balls
Obsidian
Ochre
Pyrite in rock
Quartzite
Sandstone, hard absorbent
Sandstone, soft
Shale
Shale, hard
Shale, opaline cherty
Shale, soft
Shale, some
Shell
Slate
Sulfates—sulfides, iron, in rock
Thin or elongated pieces
Unsound pieces

TABLE III.—STATE SPECIFICATION REQUIREMENTS FOR SOFT PIECES.

Item		Specification Limit, per cent	Number of Specifications
ft pieces		2	2
		3	3
		5	4
		10	1
		Free from excess	1
ft pieces, bituminous aggregate		1	2
ft pieces, concrete aggregate		2	2
		3	1
ft pieces	Concrete aggregate	6	1
	Cover material	7	
	Oil mix material	12	
	Stabilized material	20	
ay lumps		0.5	1
ale		2.0	
gr. less than 1.95		2.0	
tal shale, coal, clay lumps, and soft fragments		5	1
loss in Na_2SO_4 test is less than 2 per cent, soft shale may ot exceed		10	1
loss is 2.0 per cent or greater, soft shale limited to		6	

Table III presents a compilation of the various state specifications for soft pieces. Most specifications limit the content of soft pieces of aggregate to 5 per cent or less. In a few cases, limits for specific types of soft particles are given, but usually the specifications fail to identify the materials covered by the requirement.

Table IV lists the methods of test for determining the content of soft pieces of aggregates. The methods most commonly used are visual inspection, the Los Angeles abrasion test, the scratch test, and a test using a hand hammer or compression machine. These and the other tests listed are of two types: a test in which the individual particles are examined separately; and a test in which the effect of soft pieces on a characteristic of the entire material is determined. It should be noted that, with the exception of the requirement involving the use of the sodium sulfate test, all of the specification requirements shown in Table III necessitate the use of the first type of test and, in the one exception, this type of test is used in part. Consequently, it appears that determinations of the content of soft pieces should be based on

TABLE IV.—METHODS OF TEST FOR IDENTIFYING SOFT PIECES AND NUMBER OF STATES USING METHOD.

Method	Number of States
Los Angeles abrasion test	10
Breakage test under roller	1
Visual inspection	14
Scratch test	4
Hand hammer or compression test	4
Douglas stonemeter	2
Specific gravity	2
Solubility in acid	1
Sulfate soundness test	2
Absorption test	1
Flotation by heavy liquid	1
Gravel impact test	1
Deval abrasion test	1

actual count or weight of particles rather than on the use of an indirect method involving some characteristic pertaining to the whole sample.

As listed in Table IV, several states use certain methods of test, the Los Angeles abrasion test for example, for determining the presence of soft pieces in aggregate. However, these states fail to include numerical values in their reported requirements for soft pieces which are applicable to these methods. Although the reporting authorities may consider the methods mentioned to be suitable for use, it is probable that lack of sufficient test data has so far prevented the establishment of specification requirements.

Eighty-four samples of aggregates were submitted by the states in response to our request for typical samples of material composed of soft pieces. In a number of cases the samples were confined, as requested, to a given type of material. However, many samples were found to be composed of a number of kinds of material differing as greatly as limestone and gneiss. It is possible that our request for samples composed wholly of soft pieces was misunderstood, and that some samples were submitted which contained a small amount of soft pieces naturally occurring in these materials.

From the varieties of materials submitted as soft pieces, it is apparent that there is no concordance of opinion as to the kind of material which should be thus described. It is further apparent that the term, "soft pieces," is used as a catch-all description of a number of different types of possibly undesirable material in aggregates. Before spending much time in testing materials, some rational conception must be had of the type of material to be identified by the test procedure. Choice must be made between the application of the term, "soft pieces," to pieces of aggregate which are actually soft, or to pieces of a wide variety of characteristics. Although these latter may be undesirable for use in construction, this is their only common feature. As shown in the samples submitted, these pieces may be hard or soft, tough or brittle, sound or unsound; in fact, they include the whole gamut of physical properties of aggregates. If our tests are to identify soft pieces, the only question to be considered is whether the piece under test is hard or soft. During the course of this investigation, tests foreign to a strict hard-or-soft determination were made, whereby it was hoped that some correlation between the hardness of the particle and some other characteristic of interest could be established. As a general rule, the results were rather disappointing. In some cases with selected samples we did find definite correlations between the hardness of a material and some other characteristic, but when a wider variety of samples was considered, the agreement between the two characteristics appeared to be largely a matter of chance.

METHODS OF TEST

The tests made in this investigation may be grouped in the following classifications:

Visual inspection,
Scratch hardness,
Specific gravity and absorption,
Resistance to impact,
Resistance to abrasion,
Resistance to static loads, and
Soundness.

In some cases, the tests were made on the material as received, that is, without preliminary separation into individual sizes. In most tests, however, the size of the piece affected the test result to a marked extent, and it was necessary to sieve the test sample into separate sizes. The sizes generally used were 2 to $1\frac{1}{2}$, $1\frac{1}{2}$ to 1, 1 to $\frac{3}{4}$, and $\frac{3}{4}$ to $\frac{1}{2}$ in. Pieces smaller than $\frac{1}{2}$ in. were found to be difficult to test by some methods, and it was deemed sufficient, for the present at least, to study methods of testing the larger pieces only.

In the first series of tests made on the 84 samples submitted by the state highway departments, the samples were separated into three groups with respect to hardness of the material: soft, hard, and borderline. This was done by a combination of visual inspection and the use of a scratch test employing a steel knife. It was realized that these methods are not dependable for separating any and all materials into hard and soft classifications; however, they can be used to separate unquestionably soft material from unquestionably hard material. The materials which were found to be neither hard nor soft were classified as borderline materials and excluded from further tests.

Visual inspection of aggregates for the presence of soft pieces is associated with what might be called the luster or appearance by reflected light, and the degree of bonding. Pieces of aggregate with a glassy or stony luster are usually classed as hard; those with a dull or earthy appearance are classed as soft. Compact materials are usually placed in the hard classification, but those which have a loose or friable texture are considered as soft.

Specific Gravity and Absorption

Tests for bulk specific gravity were made on saturated and surface-dry samples using the mason jar pycnometer.[3] This determination does not appear to have any significance in differentiating between hard and soft pieces. Seven samples of hard materials had bulk specific gravities varying from 1.62 to 2.55; five samples of soft materials varied from 1.48 to 2.57; eight samples classed as borderline materials varied from 2.11 to 2.59. With so much overlapping of test values, use of the test for specific gravity to identify hard and soft pieces does not appear feasible.

The absorption test was made by immersing oven-dried samples in water for a period of 24 hr. The samples were then surface-dried with a towel and weighed, and the absorption expressed as a percentage of the dry weight. A summary of the results obtained is given in Table V. In this table, as in others to follow, each size of each material tested is treated as an individual sample. This permits comparisons between the two types of material, hard and soft, for each size of piece. Although the average values for the two types of material differ markedly, there is some overlapping in the test values. In the 1 to $\frac{3}{4}$-in. size, for example, one sample of soft material has an absorption of only 3.2 per cent, whereas a sample of hard material has a quite high absorption of 11.3 per cent. Because of this overlapping, it is not possible to set a value separating hard from soft material and

TABLE V.—USE OF THE ABSORPTION TEST FOR THE IDENTIFICATION OF HARD AND SOFT MATERIALS.

Size of piece, in.	2 to 1½	1½ to 1	1 to ¾	¾ to ½
SOFT MATERIAL				
Number of samples	4	30	31	20
Absorption, per cent				
Minimum	2.6	2.6	3.2	4.3
Maximum	23.4	30.1	29.0	30.9
Average	11.5	12.8	14.4	18.2
HARD MATERIAL				
Number of samples	6	22	22	10
Absorption, per cent				
Minimum	1.4	0.5	0.6	1.0
Maximum	11.1	11.3	11.3	11.0
Average	4.7	2.7	3.0	3.3

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[3] Described in "Principles of Highway Construction as Applied to Airports, Flight Strips, and Other Landing Areas for Aircraft," *Public Roads Administration*, June, 1943, p. 297.

this method is of little use for the identification of soft particles.

IMPACT TESTS

Several different types of impact tests were used. The one believed to have the most promise is the test for the toughness of gravel.[4] As shown in Fig. 1, the apparatus used in this consists essentially of a 2½-in. steel ball mounted on a steel block to serve as an anvil, and another steel ball of the same size which can be lifted to a maximum height of 7 in. and allowed to fall on the specimen under test. During the course of this investigation, certain improvements in the original apparatus were made so that the ball could be dropped the exact distance desired with but one measurement of the thickness of the specimen under test.

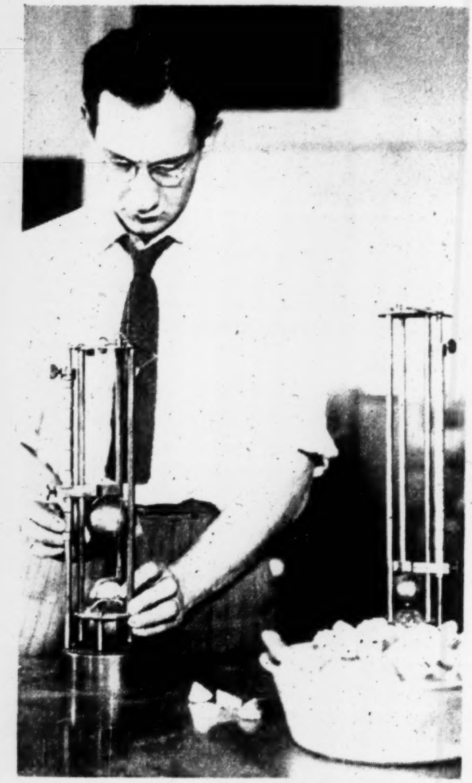

FIG. 1.—Toughness Test (2½-in. Ball) for Gravel.

In making this test, the specimen was held on the anvil in its most stable position, usually with the least dimension vertical, and the movable ball dropped on the specimen from a height of 1 in. The height of fall was increased 1 in. after each blow until the specimen failed or until it withstood a drop of 7 in. Under normal conditions, a test sample containing 50 to 100 pieces of the same sieve size was used. An empirical value for each sample was obtained by multiplying the number of pieces which failed at each successive height of drop, by the square of the drop, in inches, and dividing the sum of these values by the total number of pieces tested. For the purpose of this computation, pieces which did not fail at a drop of 7 in. were assumed to fail at a drop of 10 in. This value, called the toughness factor, can vary from a minimum of 1 to a maximum of 100.

TABLE VI.—USE OF THE TOUGHNESS TEST (2½-IN. BALL) FOR THE IDENTIFICATION OF HARD AND SOFT MATERIALS.

Size of piece, in.	2 to 1½	1½ to 1	1 to ¾	¾ to ½
SOFT MATERIAL				
Number of samples	3	28	29	20
Toughness factor				
Minimum	18.0	2.7	2.0	1.0
Maximum	59.0	46.2	16.9	5.7
Average	41.6	20.5	7.9	2.5
HARD MATERIAL				
Number of samples	7	23	23	10
Toughness factor				
Minimum	5.0	4.5	2.9	1.1
Maximum	79.0	67.2	32.3	10.3
Average	42.3	29.8	11.4	3.8

The results of these tests are shown in Table VI. Although higher toughness factors were found for the hard materials than the soft, there is a considerable amount of overlapping of the two sets of values. It is apparent that the toughness test for gravel does not separate hard from soft material. It appears that the falling ball is much too heavy to separate soft pieces from hard but brittle pieces. Furthermore, when the brittle piece fails, the fracture is apparent, but many pieces of soft material may fail without the break being seen from above as the operator would normally view the specimen. This results in the soft piece receiving a higher rating than it should have, thereby decreasing the value of the test.

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[4] Method T-6, American Assn. of State Highway Officials Book of Standards, 1938, p. 152. This method has been withdrawn by the Association.

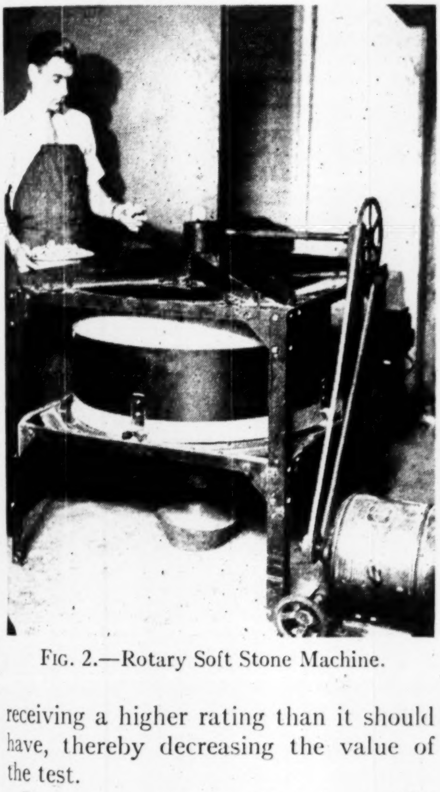

FIG. 2.—Rotary Soft Stone Machine.

In an attempt to correct these difficulties, another toughness apparatus was made using a steel ball of $1\frac{7}{8}$-in. diameter. For certain sizes of particle, the $1\frac{7}{8}$-in. ball was found to furnish more indicative test results than the $2\frac{1}{2}$-in. ball, but the conclusion was reached that to test aggregate of a complete range in size from 2 or $2\frac{1}{2}$ to $\frac{3}{8}$-in., at least three toughness testers of different sizes should be used, and each size used to test material of a definite and narrow range in sieve sizes. As time to develop these tests and correlate the test results of each was not available, further consideration of the use of this type of apparatus was deferred.

Rotary Soft Stone Machine:

Another type of impact test tried involves the use of the rotary soft stone machine shown in Fig. 2. This machine consists essentially of a cast iron disk revolving in a horizontal plane inside a vertical steel drum made of $\frac{3}{8}$-in. plate. The disk is 29 in. in diameter and the drum has an inside diameter of $33\frac{1}{4}$ in. Ribs on the upper surface of the disk form pockets to catch the material fed on the disk through a sheet metal cone, and to throw these pieces against the steel drum. Another cone below the disk serves to collect the sample and lead it to a pan in which the tested material can be inspected. The disk is powered by a variable speed motor and can be operated at speeds from 110 to 200 rpm. In these tests, the fastest speed was used. The test was conducted on individual sizes of aggregate. The weight of the test

TABLE VII.—USE OF THE ROTARY SOFT STONE MACHINE FOR THE IDENTIFICATION OF HARD AND SOFT MATERIALS.

Size of piece, in.	2 to 1½	1½ to 1	1 to ¾	¾ to ½
SOFT MATERIAL				
Number of samples	3	25	28	18
Loss, per cent				
Minimum	13.5	2.5	2.5	1.4
Maximum	22.0	29.9	21.4	20.8
Average	19.0	11.2	10.4	9.6
HARD MATERIAL				
Number of samples	5	19	21	9
Loss, per cent				
Minimum	1.0	2.0	2.5	1.4
Maximum	24.0	19.5	32.0	34.2
Average	15.9	10.3	9.9	11.6

sample was determined and the pieces passed singly through the machine. After all pieces had been tested, the sample was sieved on the original retaining sieve, and the weight of material passing this sieve expressed as a percentage of the original weight of the sample.

The results obtained with the rotary machine are given in Table VII. These results indicate the same conditions as were found for the falling ball apparatus. Hard but brittle material as well as soft material is readily broken in the rotary machine, and it does not appear possible to separate truly soft pieces from others with this type of test.

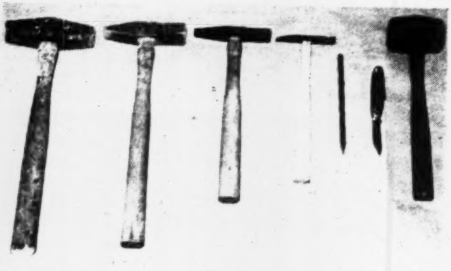

FIG. 3.—Hammers and Scribes Used in Soft Piece Test.

Left to right: 2-lb. stone mason's hammer, 1-lb. tinner's hammer, 8-oz. tinner's hammer, 2-oz. tile setter's hammer, brass scratch pencil, knife, and rubber mallet.

Hand Hammer Tests:

Several other types of impact tests were tried. In former years, a hammer test was occasionally used to identify soft pieces. Although the application of a hand hammer involves a number of indefinite conditions, it was believed desirable to include this test in the investigation. The hammers used varied from a 2-oz. tile setter's hammer to a stone mason's hammer with a 2-lb. head as shown in Fig. 3. Several different types of technique were used. In one, the hammer was allowed to drop without bending of the wrist; in another, the hammer was swung by movement of the wrist only; in a third, the end of the hammer handle was placed on the table and the hammer head allowed to fall through an arc of about 8 in. The tile setter's hammer has one flat and one sharply beveled end. With this hammer, one method included a free swing of the arm, with the flat face striking the piece under test. In another application of the same small hammer, the beveled end was used to peck at the test specimen to determine whether the material could be flecked away or cut by light taps. Consideration was also given to the type of failure of a specimen under blows of a reasonably heavy hammer. It was thought that if the material were soft, it would crush under the hammer, producing a large amount of powder with relatively few large fragments. Hard material, on the other hand, would break to sharp-edged fragments with relatively little powdering. Of all these tests, none gave satisfactory results, and this type of test was discontinued.

Consideration was then given to an impact test using as the striker an article which would deform around the piece under test but still transmit stress to the piece. The articles considered for this application were a device known in some circles as a blackjack, and a mallet with a rubber head such as is used in removing dents from automobile bodies. A blackjack having a leather case filled with lead shot was tried, but after a few tests the leather broke and further consideration of this was discontinued. For a few very soft materials, the rubber mallet gave satisfactory results, but moderately soft materials of 1-in. size or larger could not be crushed with the mallet. This test was also discontinued.

COMPRESSION TESTS

The suitability of a compression test to identify soft pieces was tried. A small apparatus[5] for such a test has been used, but the maximum load specified is only 75 lb. and this load will crush only the weakest specimens. This apparatus is shown in Fig. 4. The use of an hydraulic compression testing machine was then proposed. In the method considered, an attempt was made to apply load to the particle through ½-in. steel balls. For pieces of irregular shape, this was very difficult to perform, and flat-faced steel cylinders of ½-in. diameter were substituted for the balls. The pieces under test were ground on a lap to secure bearing faces, and were tested with the least dimension in a vertical position. Load was applied at a slow but predetermined rate and failure determined to the nearest 10 lb. Each sample tested contained from 20 to 100 pieces.

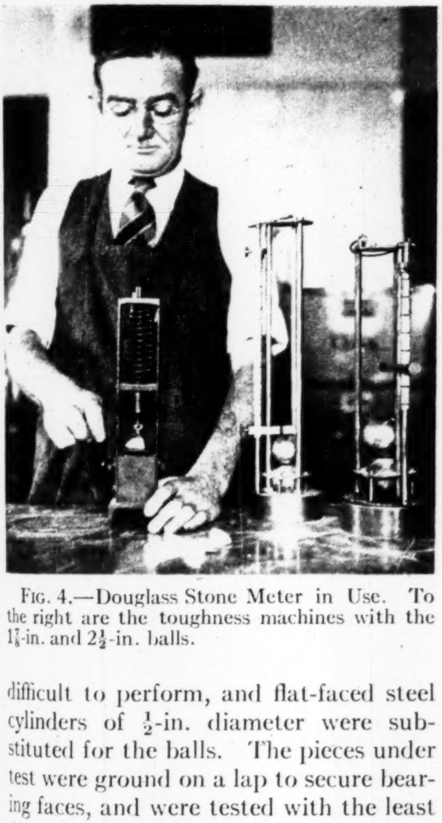

FIG. 4.—Douglass Stone Meter in Use. To the right are the toughness machines with the 1⅞-in. and 2½-in. balls.

The results obtained are given in Table VIII. It will be observed that no well-defined separation between hard and soft materials is obtained. Study of the pieces under load revealed one interesting feature. In testing a shale which occurs in rather thin pieces, it was noticed that the portion of the piece not in contact with the loading cylinders fell away at a relatively low load, but the material between the loading cylinders became more compact as the loading continued. In one case, this material was loaded to 35,000 lb. or almost 180,000 psi. It is apparent that this type of loading is unsuitable for thin specimens of soft material and that the test does not separate hard from soft material.

TABLE VIII.—USE OF THE COMPRESSION MACHINE FOR IDENTIFICATION OF HARD AND SOFT MATERIALS. LOAD APPLIED THROUGH FLAT FACES OF ½-IN. DIAMETER.

Size of piece, in.	2 to 1½	1½ to 1	1 to ¾	¾ to ½
SOFT MATERIAL				
Number of samples	4	9	20	8
Compressive strength, lb.				
Minimum	1130	540	120	120
Maximum	2910	2970	2770	3530
Average	1840	1270	860	1010
HARD MATERIAL				
Number of samples	5	10	13	3
Compressive strength, lb.				
Minimum	500	860	690	480
Maximum	3460	2950	3250	1290
Average	1890	1830	1750	860

A subsequent restudy of the data indicated that possibly a fair trial had not been given to the compression test. The labor of grinding faces on 50 to 100 pieces of each size in each sample tested was considered objectionable, and some means of testing pieces of any shape without preliminary grinding was sought. Trials were made of a number of different types of loading devices. After considerable work, a loading device consisting of a single point upper contact and a three point lower support was adopted. As shown in Fig. 5, the upper contact consists of a short steel rod of ½-in. diameter, with a hemispherical end, which is fastened to the spherical bearing block of an hydraulic cube-testing machine. The lower bearing consists of three ½-in. diameter steel balls grouped together so that their surfaces are in contact, and welded to a small steel base. With this loading and support arrangement, most pieces of both regular and irregular shape can be placed in a stable position for the test.

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[5] Douglass stone meter described in Method T-8, American Assn. of State Highway Officials Book of Standards, 1938, p. 154. This method has been withdrawn by the Association.

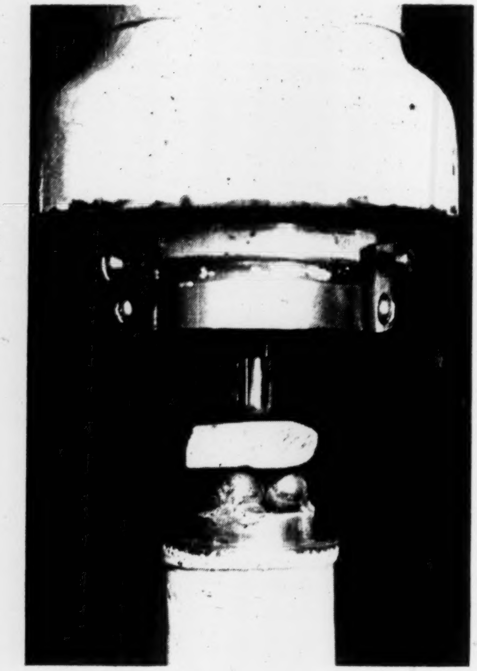

FIG. 5.—Compression Test for Soft Pieces. Three point support and single point load.

To obtain values for use in identifying pieces of hard, sound aggregate, several hundred pieces of each size of three different materials were tested and the breaking load of each piece determined. From the data obtained, the following loads were selected as indicating hard materials:

Size of Piece, in.	Load, lb.
⅜ to ½	200
½ to ¾	350
¾ to 1	500
1 to 1½	750
1½ to 2	1100
2 to 2½	1500

Based upon these tentative values for hard material tests were carried out in conjunction with certain other tests, as described below under Tests of Commercial Gravels.

FREEZING-AND-THAWING TEST

The freezing-and-thawing test was used to determine whether any relationship could be established between the results of this test and the hardness of the materials considered. The samples were immersed in water for 24 hr. prior to freezing, frozen at about 15 F. in water and thawed at about 80 F. A 24-hr. cycle was used. After ten cycles of the test, the samples were examined. With only a few exceptions, this short test failed to differentiate between hard and soft materials. The test was then resumed for another ten cycles. After

TABLE IX.—USE OF FREEZING AND THAWING (20 CYCLES) FOR IDENTIFICATION OF HARD AND SOFT MATERIAL.

Size of piece, in.	2 to 1½	1½ to 1	1 to ¾	¾ to ½
SOFT MATERIAL				
Number of samples	1	12	21	11
Loss passing ½-size sieve, per cent				
Minimum		3.1	5.0	9.4
Maximum		92.8	93.0	92.2
Average	9.2	26.1	42.5	51.5
HARD MATERIAL				
Number of samples	4	11	14	3
Loss passing ½-size sieve, per cent				
Minimum	2.7	0.6	0.4	5.4
Maximum	31.6	18.2	51.4	25.3
Average	11.8	5.0	11.4	13.1

the twentieth cycle, the samples were dried, sieved on a sieve with openings having linear dimensions one-half the size of those in the original retaining sieve, and the material passing the sieve expressed as a percentage of the weight of the original sample. The half-size sieves were used rather than the original sieve to prevent the inclusion in the loss of those pieces which might pass the original retaining sieve due only to minor flaking or chipping. The results of these tests are given in Table IX.

TESTS OF COMMERCIAL GRAVELS

For further tests, the National Sand and Gravel Assn. was requested to furnish samples of commercially produced gravels which would contain some soft material, since the material supplied by the states was almost exhausted. To the samples received from the Association were added a number of gravels which had been submitted to the laboratory for routine tests. These samples were tested for soft piece content using the compression test described above, the 20-cycle freezing-and-thawing test, and the Los Angeles abrasion test. In the last test, determinations of the percentage of wear were made at 100 and 500 revolutions and a ratio of the losses used as an expression of the amount of soft material in the sample.

The results of these tests are given in Table X. Inspection of the results discloses immediately that there is little agreement between the indications of the three methods of test. For some few samples, all three methods show the presence of appreciable amounts of soft material. In most cases, however, the three methods give different results. One reason for nonuniformity among the three methods is the unit of failure on which the test result depends. In the compression test, the pieces of the sample are tested separately, and the whole piece is discarded if it fails to meet the conditions of the test. In the Los Angeles abrasion and freezing-and-thawing tests, it is entirely possible that only a portion of a given particle may be included in the loss. Consequently, it is doubtful that any satisfactory agreement among different methods of test can be established unless the unit of failure is the same in each case.

TABLE X.—SOFT PIECE TESTS ON COMMERCIALLY PRODUCED GRAVELS.

Sample	Los Angeles Abrasion Test			Compression Test, per cent soft by weight	Freezing-and-Thawing 20 Cycles, Loss, per cent
	Loss at 100 Revolutions, per cent	Loss at 500 Revolutions, per cent	Ratio of Losses, 100/500, per cent		
No. 67464...	6.0	28.1	21.4		27.4
No. 67621...	4.9	24.2	20.2		10.4
No. 67622...	4.4	22.7	19.4		10.3
No. 67628...	8.1	32.6	24.8		9.1
No. 67629...	5.4	27.3	19.8		9.3
No. 67680...	4.2	23.4	17.9		6.8
No. 67696...	4.6	23.8	19.3		7.5
No. 67757...	5.6	23.6	23.7	12.1	4.2
No. 67858...	7.3	26.4	27.7		11.0
No. 67909...	5.6	23.3	24.0		17.0
No. 67910...	4.2	25.0	16.8		17.2
No. 67954...	6.1	27.8	21.9		20.4
No. 68516...	5.6	26.5	21.1	15.6	11.2
No. 68768...	7.6	30.7	24.8	24.5	19.7
No. 68769...	5.4	26.3	20.5	19.5	6.5
No. 68770...	5.7	27.1	21.0	21.3	5.7
No. 68771...	4.1	21.3	19.2	14.4	7.6
No. 68772...	6.5	29.0	22.4	26.2	8.4
No. 68773...	5.3	26.6	19.9	23.7	3.8
No. 68774...	8.5	32.9	25.8	33.1	12.7
No. 68775...	6.8	30.8	22.1	18.8	8.9
No. 68776...	6.4	31.8	20.1	38.9	21.3
No. 68777...	7.4	32.4	22.8	29.8	29.2
No. 68778...	6.8	29.6	23.0	14.0	4.1
No. 68779...	5.8	25.5	22.8	23.7	2.4
No. 68780...	6.6	30.3	21.8	17.6	9.5

Consideration of the values obtained in the compression test shown in Table X indicates that the limits set for the identification of hard pieces are probably too severe. A review of the individual test results for each sample shows a very high percentage of failures in the smaller sizes of piece and also an excessive percentage of failures for pieces which tend toward a flat shape. In this test, the point of application of the load is normally above a point equidistant from the point of support of each of the three balls in the lower bearing. With thin or flat pieces, this could result in flexure of the piece under test, and the failure would be by bending instead of by compression.

REVIEW OF METHODS OF TEST

Late in 1946 several new methods of testing aggregates for content of soft pieces were suggested. These included a scratch test using a scribe of yellow brass, and two abrasion tests. In one of these abrasion tests, the sample was placed in a canvas bag with a charge of steel balls and the bag dropped on or swung against an anvil a given number of times. In the other test, the sample alone was placed in a bag and subjected to blows delivered by a rubber-headed mallet, freely swung as shown in Fig. 6. In trying these methods, it was considered desirable to include in the program of tests some of the methods which had previously been used.

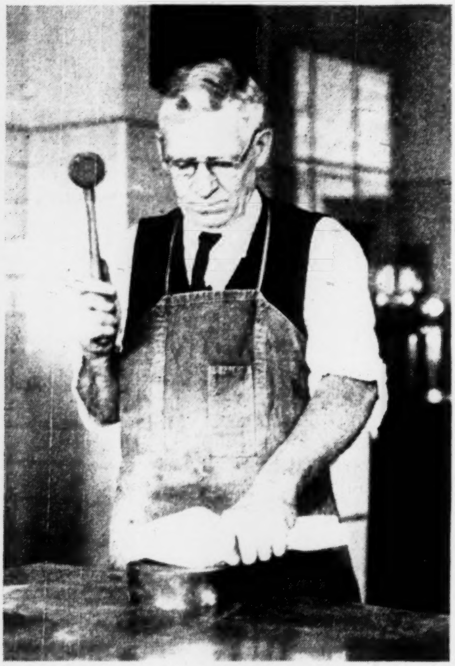

FIG. 6.—Bag and Mallet Test for Soft Pieces.

The material used in these tests consisted of quartz gravel of a reasonably uniform hardness to which was added definite quantities of soft stone or gravel obtained from the remnants of the samples received in 1941. Objection was made that the personnel would be influenced by the color of the different pieces and would unconsciously classify pieces on this basis. Consideration was given to the possibility of dyeing all the material to be used to a uniform color, or fitting the operators with colored glasses. Both suggestions were rejected and the difficulty overcome in part by the use of some soft material of nearly the same color as the base gravel. Furthermore, most of the tests made have definite end points, and the operator is not required to decide whether the material does or does not meet the conditions of the test.

Several changes in the test procedure as previously used were thought to be desirable. A brief description of each method used is given below:

Method 1:

Steel scratch test. Each piece was scratched with a sharp knife blade. The weight of the pieces identified as soft was reported as a percentage of the original weight of the sample.

Method 2:

Brass scratch test.—Each piece was scratched with a pointed scribe prepared from $\frac{1}{4}$-in. diameter yellow brass rod. The results were reported as in Method 1.

Method 3:

Light hammer test.—The test piece was placed on an anvil in its most natural position of repose, held firmly with the fingers, and struck at the center of the upper face with the flat end of the tile setter's hammer. The hammer was swung through an arc of about 6 in. using a natural movement of the wrist only. Softness was recognized by crushing or crumbling of the piece under test. A clean fracturing or splintering of the piece, that is, breaking into smaller but solid fragments without considerable powdering, was not considered indicative of softness. More than one blow was permitted if necessary to classify the piece properly. The results were reported as in Method 1.

Method 4:

Rubber mallet test.—The method followed the procedure given in Method 3.

Method 5:

Toughness test for gravel.—The $2\frac{1}{2}$-in. ball was dropped as follows:

Size of piece, in.	Height of fall, in.
$1\frac{1}{2}$ to 1	3
1 to $\frac{3}{4}$	2
$\frac{3}{4}$ to $\frac{1}{2}$	1

The test piece was held on the anvil and adjusted under light tapping of the free ball to a secure bearing. The free ball was then raised to the height indicated for the size of aggregate under test and allowed to fall on the piece. Softness of the piece was shown by crushing, powdering, or crumbling. Each sample was reported as in Method 1.

Method 6:

Toughness test for gravel.—The procedure given in Method 5 was repeated using the apparatus containing the $1\frac{7}{8}$-in. ball, except that the heights of fall were:

Size of piece, in.	Height of fall, in.
$1\frac{1}{2}$ to 1	5
1 to $\frac{3}{4}$	3
$\frac{3}{4}$ to $\frac{1}{2}$	1

Method 7:

Rotary soft piece test.—The machine was operated at its slowest speed (about 110 rpm.). The pieces of the test sample were fed separately into the machine and all debris caught. The material passing the original retaining sieve was determined, and expressed as a percentage, by weight, of the original sample.

Method 8:

Douglass stone meter test.—The following loads were used:

Size of piece, in.	Applied load, lb.
$1\frac{1}{2}$ to 1	125 (Maximum loading of spring)
1 to $\frac{3}{4}$	75
$\frac{3}{4}$ to $\frac{1}{2}$	60

The results were reported as in Method 1.

Method 9:

Compression test.—The tests were made in the hydraulic testing machine, using the following loads:

Size of piece, in.	Applied load, lb.
$1\frac{1}{2}$ to 1	500
1 to $\frac{3}{4}$	350
$\frac{3}{4}$ to $\frac{1}{2}$	200

The piece tested was placed in its most stable position on the three point support, and load applied without shock through a single point contact on the upper surface of the specimen. Breakage of the specimen at a load below those indicated constituted failure. The results were reported as in Method 1.

Method 10:

Bag abrasion method.—A 1000-g. sample was placed in a 10 by 14-in. canvas bag with an abrasive charge of five $1\frac{7}{8}$-in. diameter cast iron or steel balls. The neck of the bag was fastened securely to prevent loss of the sample. The bag was swung 100 times through a distance of 12 in. against an anvil. The sample was then removed from the bag and sieved on a No. 4 sieve. The material passing this sieve was expressed as a percentage of the original weight of the sample.

Method 11:

Bag and mallet method.—A 1000-g. sample was placed in a 6 by 9-in. canvas bag and the neck fastened securely to confine the sample in the least space. The bag was then placed on an anvil and struck 100 times with a rubber-headed mallet. The blows were distributed over the side of the bag, and after each tenth blow the bag was turned over to expose the lower side. The results were determined as in Method 10.

TABLE XI.—RESULTS OF TESTS FOR SOFT PIECES USING PREPARED SAMPLES.

Size of Piece, in.	Soft Pieces, per cent by weight 1½ to 1	1 to ¾	¾ to ½	Average
Soft pieces placed in material	4.8	16.2	10.4	10.5
SCRATCH HARDNESS METHODS				
By steel scribe	17.6	18.2	14.9	16.9
By brass scribe	4.8	16.3	10.6	10.5
IMPACT METHODS				
2-oz. hammer	11.3	18.8	17.9	16.0
Rubber mallet	1.4	10.1	12.6	8.0
Rotary machine	14.6	10.6	10.9	12.0
Toughness 2½-in. ball	10.2	34.1	21.1	21.8
Toughness 1⅞-in. ball	20.1	22.0	10.2	17.4
COMPRESSION METHOD				
Douglass machine	8.5	4.8	0.6	4.6
Three-point support	4.2	16.9	21.0	14.0
ABRASION METHOD				
Bag abrasion	6.7	18.0	14.0	12 9
Bag and mallet	6.4	11.7	11.7	9.9
Los Angeles machine				
Loss at 100 revolutions	5.1	11.9	14.8	...
Loss at 500 revolutions	22.5	40.3	45.3	...
Ratio 100/500	22.7	29.5	32.7	...

Method 12:

Los Angeles abrasion test.—Samples weighing 5000 g. of each size of material were tested with an abrasive charge of twelve steel balls weighing 5000 g. The percentage of wear was determined at 100 and 500 revolutions.

DISCUSSION OF TEST RESULTS AND METHODS OF TEST

The results obtained in these tests are given in Table XI. The values shown at the top of the table are the percentages by weight of soft material which were added to the quartz gravel. The values given in the rest of the table, except for those of the Los Angeles abrasion tests, are the percentages by weight of soft material found by the test method indicated. The values given for the Los Angeles tests are the losses in percentages by weight obtained in the tests. The ratio of the losses at 100 and 500 revolutions has been thought to have some relation with the amount of soft material in the sample. A comparison between these values and the amounts of soft material actually added to the base gravel indicates that this test as made on individual sizes of aggregate, does not furnish test results which are indicative of the amount of soft material in the sample.

The most favorable results obtained in this series of tests are those furnished by the brass scribe, the rotary soft piece machine, and the bag and mallet test. In previous work, when the rotary machine was operated at a speed of 200 rpm., it had not given very significant results. Although much better results are obtained when the machine is operated at a slower speed, further consideration of this type of testing equipment was discontinued as the machine is not suitable for field use.

Bag and Mallet Test:

Some question developed regarding the size of sample which would be most desirable in the bag and mallet method, and also how many tests should be made to obtain a test value representative of a given material. From a practical consideration, a sample of the size used appears to be about as large as is desired for readiness of handling. Should the material to be tested have a maximum size greater than 1½ in., the size of the test sample must be increased and a larger bag used. Considerable development work along this and other lines remains to be done before this method can be adopted for use. To determine the other feature of particular interest here, that is, the number of 1000-g. samples which should be tested for a material with a maximum size of 1½ in., another series of tests was made.

TABLE XII.—BAG AND MALLET TESTS ON PREPARED SAMPLES OF QUARTZ GRAVEL CONTAINING SOFT SANDSTONE.

Sample	0 per cent Soft Stone		4 per cent Soft Stone		8 per cent Soft Stone		12 per cent Soft Stone	
	Loss, per cent	Cumulative Average Loss, per cent	Loss, per cent	Cumulative Average Loss, per cent	Loss, per cent	Cumulative Average Loss, per cent	Loss, per cent	Cumulative Average Loss, per cent
No. 1	4.4	4.4	6.2	6.2	12.5	12.5	9.7	9.7
No. 2	3.7	4.0	6.2	6.2	9.9	11.2	11.2	10.4
No. 3	5.9	4.7	6.3	6.2	8.7	10.4	12.3	11.1
No. 4	4.3	4.6	7.4	6.5	10.6	10.4	10.7	11.0
No. 5	3.7	4.4	5.8	6.4	8.2	10.0	13.1	11.4
No. 6	3.9	4.3	7.0	6.5	9.0	9.8	12.4	11.6
No. 7	4.8	4.4	6.0	6.4	10.0	9.8	13.4	11.9
No. 8	4.7	4.4	6.3	6.4	6.9	9.5	9.8	11.6
No. 9	5.0	4.5	6.5	6.4	6.9	9.2	10.9	11.5
No. 10	3.7	4.4	5.0	6.3	9.5	9.2	9.1	11.3

PERCENTAGE SOFT STONE REMAINING IN SAMPLE AFTER TEST, AVERAGES FOR TEN SAMPLES

Soft Stone	
Placed in Sample, per cent	Remaining After Test, per cent
4	2.0
8	3.3
12	4.5

In these tests, a soft sandstone was added in definite quantities to a quartz gravel to prepare four aggregates containing 0, 4, 8, and 12 per cent soft material. Each material, both the hard and soft, was graded uniformly from 1½ to ⅜ in. After each aggregate had been prepared, it was mixed thoroughly and ten 1000-g. samples taken for test. The attempt made here was to duplicate conditions which would exist if a sample of graded aggregate were tested by the bag and mallet method.

A summary of the results is given in Table XII. The percentage of loss for each sample tested is given together with a cumulative average. For most aggregates, an average value obtained from tests of three samples is very nearly the same as the average for all ten samples of a kind tested. However, of more importance is the comparison between the amount of soft stone placed in the aggregate, the test result obtained, and the amount of soft stone remaining in the sample after the test. Tests of the base gravel show a loss of 4.4 per cent. The results obtained in tests of the gravel containing 4 per cent of soft stone show 6.3 per cent loss, but 2.0 per cent of soft stone is left in the sample. This 6.3 per cent loss, then, includes 2.0 per cent of soft stone and 4.3 per cent from the quartz gravel.

By a similar method of figuring, the test results for the aggregates containing 8 and 12 per cent of soft stone, include 4.5 and 3.8 per cent, respectively, of the quartz gravel in the loss. Although it is granted that the quartz gravel may contain material of a friable nature, a comparison of the gravel and the sandstone used in these tests indicates that a satisfactory method of test should show a greater recovery of the soft material in the test results. The fact that from 38 to 50 per cent of the soft sandstone placed in the sample remains there after the test shows definitely that the test as made is not satisfactory.

Brass Scratch Test:

One of the chief difficulties in the development of a satisfactory method of test for soft pieces is in trying to unify the conceptions offered by different authorities regarding a proper description of a soft piece. In addition to the various tests described here, many more fanciful tests have been considered and, in some cases, plans for quite elaborate pieces of testing equipment have been prepared. A few of these fanciful tests have been tried, but they did not prove even remotely satisfactory. Possibly we have been trying to make something difficult of a really simple problem. We are concerned with the testing of aggregates to determine the presence and quantity of soft pieces—those which yield easily to physical pressure or are not resistant to cutting or wear.

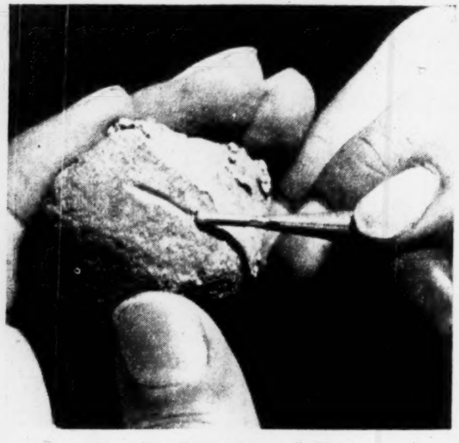

FIG. 7.—Brass Scratch Test.

In all of our efforts, the most simple and direct method is a scratch hardness test. This would not indicate the pieces which are unsound, or light-weight, or highly absorptive; or the pieces of chert which appear to be included by some authorities in the general classification of soft pieces. It would, however, show which pieces of the sample are actually soft, including those formed of a soft material and those which are so poorly bonded that the separate particles in the piece are easily detached from the mass.

A satisfactory test for scratch hardness has already been used in this investigation. It consists merely of scratching the material under test with a piece of yellow brass as shown in Fig. 7. This brass will not scratch limestone of good quality, but it is hard enough to scratch badly weathered materials which may be objectionable for use in concrete. The brass used first in these tests consisted of a $\frac{1}{4}$-in. rod which has a Rockwell hardness of about B70. Later in this work, the thought of preparing a pencil with a brass rod replacing the lead was developed. For this purpose, drill rod brass[6] of about $\frac{1}{16}$-in. diameter was used. Efforts were made to obtain a Rockwell hardness value for this material, but the rod was too small to secure a satisfactory reading. For the purpose of this test, it is probable that minute distinctions in the hardness of the brass used are of little moment. Possibly it is sufficient to describe the material as hard, yellow brass.

The test consists of separating the aggregate into different sizes down to $\frac{3}{8}$ in. and determining the scratch hardness of a representative number of pieces of each size. With material of fairly uniform quality, 10 pieces of a size may be sufficient, but 50 or 100 pieces of a size may be required for heterogeneous materials. The weight of the pieces identified as soft is determined for each size, and a weighted average based on the grading of the sample is computed.

As mentioned above, this test is for soft pieces only. If it is desired to limit the amount of other types of deleterious materials in aggregate, separate mention of these should be made in specifications.

CONCLUSION

The tests reported here were made in an attempt to develop a rational method of testing aggregates for soft piece content. Although most tests used standard or readily procurable apparatus, some rather fanciful or weird contraptions were designed or actually constructed for this purpose. On the whole, it is believed that a serious attempt has been made to study all different methods of test which could be expected to give information of value.

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[6] So called as it is obtainable in the same sizes as steel twist drills.

Of all methods tried, the only one considered suitable for laboratory and field use is the scratch hardness test using a hard, yellow brass scribe. For convenience, a pencil with a brass core is suggested.

Attention is called to the practice among some specification writers of using the soft piece classification as a catch-all for many different types of deleterious substances. If it is desired to limit types of deleterious substances other than soft pieces, these other types should be mentioned specifically and separately from pieces which are merely soft.

Acknowledgment:

The courtesy of the different state highway departments and the National Sand and Gravel Assn. in furnishing samples for use in these tests is appreciated. Mention is also made of the many valuable suggestions offered by T. R. Smith, and of the assistance furnished by him and the other employees of the Laboratories of the Public Roads Administration.

DISCUSSION

Mr. Roger H. Cook[1] *(presented in written form)*.—The following comments on the hand tool devised by Mr. Woolf for determining soft pieces in aggregate are based on the use of the tool during petrographic examination of several concrete aggregates and specimens of rock in the Petrographic Laboratory of the Bureau of Reclamation.

The tool consists of the wooden shaft of a pencil into which a brass wire with a Rockwell hardness of B69 has been inserted. The Rockwell hardness of B69 is equivalent to slightly less than 3 on Mohs' scale of hardness (the hardness of a calcite crystal). Any rock that can be scratched by the wire is presumably softer than the wire, and therefore is classified as a soft particle.

Experiments with the tool indicate that care must be exercised to be sure the particle is actually scratched and not gouged. For example, a calcite crystal (Mohs' scale hardness 3) cannot be scratched by the wire, yet by exerting a certain amount of pressure, a deep groove can be cut in the crystal. Unless the individual applying the tool is aware of this possibility, a physically sound limestone composed essentially of calcite might be classified as a soft particle. Therefore, in tests performed at the Petrographic Laboratory a rock was considered as scratched if a groove was made in the rock when the tool was drawn over the rock with a small amount of pressure, and if no metal fragments from the wire were found in the groove.

Use of the tool demonstrates that the wire will scratch claystones, argillaceous (clayey) limestones, shales, and some siltstones which are known to be unsound. These rocks are composed principally of minerals whose hardness is less than 3 on Mohs' scale. When correct criteria to establish the degree of scratching were applied, all rocks scratched by the wire are those which are actually unsound.

However, the wire would not scratch specimens of two sandstones which lost 95 per cent or more in 500 revolutions in the Los Angeles abrasion test and which would be regarded as "soft particles" according to ordinary concepts. Similarly, the wire did not scratch a ferruginous sandstone, a weathered granite, or a sandy siltstone, all of which would be regarded as soft particles in aggregate. These rock types are composed predominantly of minerals such as quartz, whose hardness is greater than 3 on Mohs' scale.

Empirically, it may be concluded that particles which are truly scratched are actually "soft" pieces. However, particles which are not scratched may be either "hard" or "soft" pieces.

These observations raise a question as to what characteristics of the particles control the degree to which they can be scratched by the wire. The degree of scratch depends straightforwardly upon the mineralogic hardness of the individual granular components of the particles being tested, whereas the degree to which the particle can be gouged depends upon the integrity of bond between the component grains. Consequently, particles composed predominantly of minerals softer than the wire will be scratched as well as gouged. Particles composed of minerals harder than the wire will not be scratched, but they may be gouged if the grains composing the particles are not firmly knit together. This last group comprises the soft sandstones which lost 95 per cent or more in the Los Angeles abrasion test. These sandstones are composed of quartz grains (hardness 7 on Mohs' scale) cemented by a small amount of quartz deposited in the intergranular spaces; therefore the sandstones are not easily scratched, but they are quickly disintegrated by abrasion and impact in the Los Angeles abrasion test. Incidentally, the abrasion test ordinarily would reveal both the soft particles scratched by the wire, and those which are not.

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[1] Petrographer, Bureau of Reclamation, Denver, Colo.

It is to be emphasized that the tool may detect a degree of softness or weakness of particles, but because softness or weakness is related only indirectly to unsoundness of aggregate, the tool cannot be expected to detect many of the particles which would decrease the quality of concrete. For example, aggregate particles contribute to reduced freezing-and-thawing durability of concrete largely because of the abundance and small size of the interconnected void spaces which they contain, not necessarily because they are weak. Thus, weathered cherts are hard and strong, yet they yield to disruption with freezing. Other particles may cause disruption of concrete because they expand with absorption of water. Thus, an altered dolerite containing about 30 per cent of the clay mineral nontronite caused rapid deterioration of concrete subjected to freezing and thawing; yet, this dolerite definitely was not scratched by the wire.

Despite the inadequacies of the scratching test, the tool would assist in the application of the petrographic examination, a procedure found to be of great value in establishing quality of aggregate. If a specification for acceptance of aggregate were to be established around the tool and test devised by Mr. Woolf, then the petrographer would have a means, other than his personal observation, of establishing the abundance of those unsound particles which are soft enough to be scratched by the wire. However, those aggregates containing significant proportions of unsound particles not scratched by the wire would not be eliminated by the test, so that other tests and determinations would be required.

On the basis of study and use of the tool devised by Mr. Woolf, it is concluded that the technique would serve as a valuable supplement to the petrographic examination of aggregates.

MR. N. T. STADTFELD.[2]—I should like to ask how one determines the Brinell or other hardness on a thin rod of that kind?

MR. D. O. WOOLF (*author*).—Tests for the hardness of the $\frac{1}{16}$-in. brass rod were made by the Engineering Mechanics Section of the National Bureau of Standards. A Vickers testing machine with a 136-deg. square based diamond pyramid indenter and a 10-kg. load was used. The three samples tested had an average hardness number of 170.

Since the preparation of this paper, scratch tests have been made on a number of aggregates using brass scribes with Rockwell hardness numbers of B64, B69, B74, and B83. No significant differences have been found between the results of the tests made with each of the four scribes.

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[2] Division Engineer, Board of Water Supply, City of New York, New York, N. Y.

UNFAVORABLE CHEMICAL REACTIONS OF AGGREGATES IN CONCRETE AND A SUGGESTED CORRECTIVE*

By W. C. Hanna[1]

"Speech may sometimes do harm; but so may silence, and a worse harm at that" is quoted from Struther (**11**).[2] The chemical reaction of aggregates in concrete has interested me for decades. As early as 1934, I revealed a severe case of unfavorable reaction which was the cause of at least part of the severe cracking in a large structure. Other data concerning the chemical reactions of aggregates in concrete have been made available by a number of persons making a study of the problems. According to Webster, aggregate is "any hard inert material for mixing in graduated fragments with a cementing material to form concrete, plaster, or the like." If this is correct then any material used in concrete which is altered by chemical reaction is not aggregate. The fact is, however, that much material is used as aggregate which is seemingly inert by most tests but which, in certain combinations, is very reactive (**2, 5, 6, 10, 12**).

Discussion of Unfavorable Chemical Reactions of Aggregates

Some of the most badly cracked concrete that I have examined have been structures where extra effort and expense actually contributed to the failures rather than preventing them. These failures may in the end help us to provide remedies so that better work may be expected.

A few figures from our work done in 1934 (**5**) will still be of interest to some since the gelatinous material, apparently responsible for at least some of the cracking, was determined to be a calcium silicate.

Figure 1 shows a brown-colored piece of aggregate in concrete together with some pockets filled with a gelatinous material which we determined as originating in the particle. Figure 2 shows the white gelatinous exudation which was produced from a questionable particle embedded in the concrete by keeping the lower part of the specimen of concrete in a pan of water so that the particle under examination was above the water. Notice that the gelatinous exudation is not in the form of a bead. Figure 3 shows cracks inside of the concrete which we found filled with gel and which we believe were caused by the unfavorable reaction between the lime water in the concrete and the aggregate under examination. It is also believed that there would be similar reaction with soluble sodium and potassium compounds which is confirmed by Stanton's (**10**) able pioneer work.

In a field search, aggregate particles were found which would react in lime water. Other investigators[3] also found such particles. As in sand-lime brick, beneficial reaction would be expected between the lime water in cement and siliceous material, but with some aggregates there is definite evidence of unfavorable results.

In 1943 some badly cracked concrete

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Chief Chemist and Chemical Engineer, California Portland Cement Co., Colton, Calif.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 999.

[3] G. W. Ward, Geologist, Portland Cement Assn., M.S. 1936.

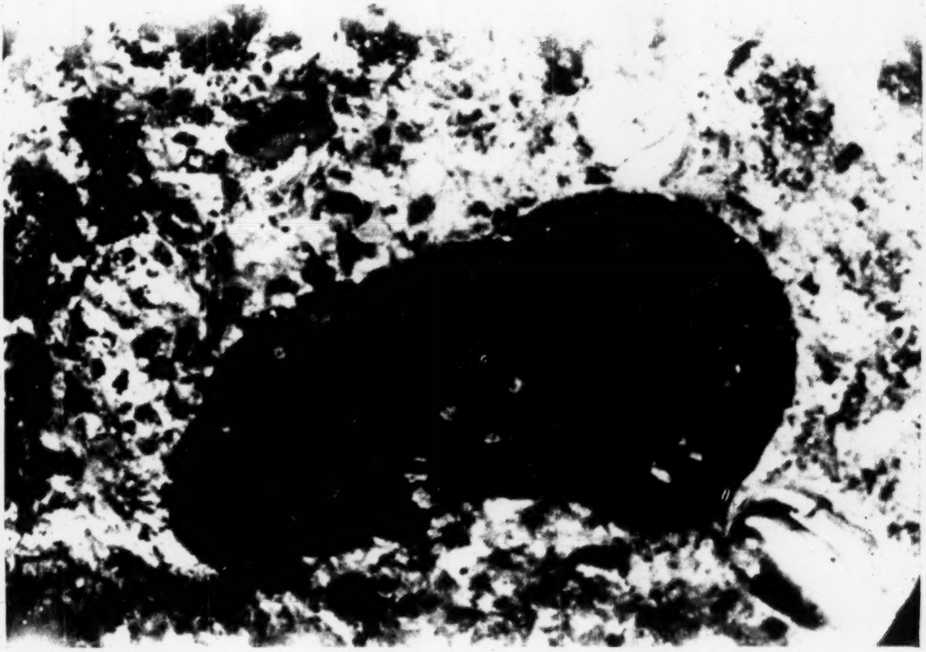

FIG. 1.—Reactive Particle (×25). Gel pockets at upper and lower right of particle. Reduced to about three-fourths size in reproduction.

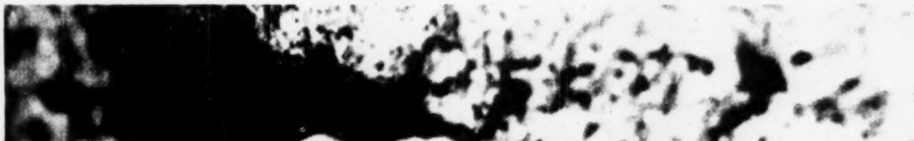

FIG. 2.—Reactive Particle and Gel (×4).

with numerous pop-outs was investigated, which had liquid, or semi-liquid, transparent globules resembling water glass, oozing from the cracks. The concrete was in a different district than had been reported upon by other investigators. The aggregate seems to have been for the most part a crushed limestone, but mixed with this were many pieces of soft brown stone which we called schist. The craters from pop-outs showed the schist at the bottom and in some cases (Fig. 4) the pop-out adhered to the liquid which finally became hard. There were so many of these globules that it was an easy matter to select plenty of clean specimens for chemical analysis. The concrete was at least fifteen years old and had often

TABLE I.—CHEMICAL ANALYSIS OF "BEADS" FROM CRACKED CONCRETE AND POP-OUTS.

Constituent	Per Cent
SiO_2	50.00
Al_2O_3 and Fe_2O_3	0.50
CaO	2 75
MgO	0.15
SO_3	2.59
Loss on ignition	11.04
Na_2O	24.67
K_2O	8:16
Cl	Trace
Total	99.86

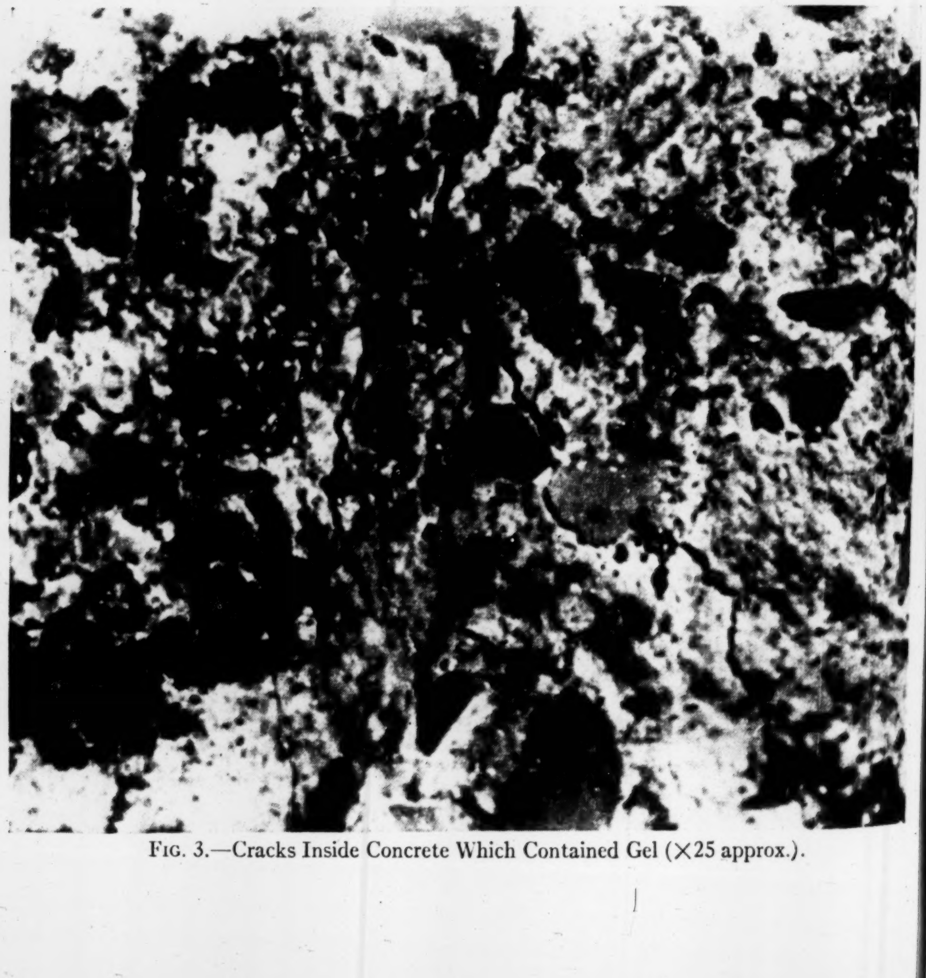

FIG. 3.—Cracks Inside Concrete Which Contained Gel (×25 approx.).

been subjected to rather high temperatures and humidity. The particles of gel varied from soft and sticky, through soft and nonsticky, to hard. Most of them were used for analysis. They were heated to approximately 225 C. for about 2 hr. They foamed and fused but became hard on cooling and were ground for analysis with the results given in Table I.

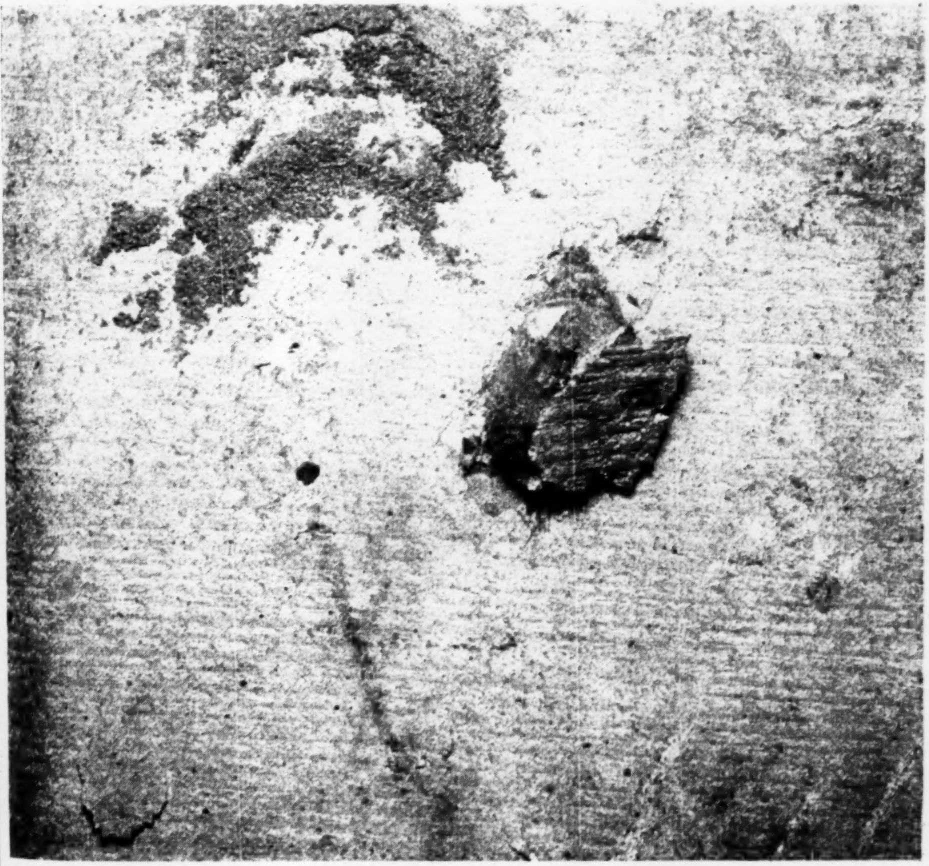

FIG. 4.—Bead of Gel with Popped-Out Concrete on Surface. At lower left another pop-out. (×2 approx.)

Our analyses of exudations indicate a wide variation in the composition of the product of uncontrolled chemical reactions of the aggregates. Tremper (12) showed an analysis of gel in which there was 85 per cent of SiO_2, and Kalousek (7) gives us the composition of a number of these gels, indicating that there is a far from constant product from these reactions.

Blanks and Meissner (2) cite analysis of water from the bottom of mortar bar containers showing a variance in composition. This indicates to me that the concretes could also contain solutions of different compositions. Bates (1) reported that waters pressed from pastes of cements were highly saturated at least with respect to the lime and to other oxides, variable in composition, and so highly alkaline that in many cases the glass was etched. Others have done work that has confirmed these data.

The following quotation from Bates (**1**, p. 47) is of particular interest because of his wide experience with cements and concretes: "When we consider what a terrible hodgepodge portland cement is, and realize what a terrible mixture all kinds of aggregates are, should we wonder that the resulting concrete may at times be surprising. Then, we have used additions to cement and to concrete over a long period of years, and too frequently they are just what a good salesman is able to put across. Then remember that we use water which may come from anywhere under or over the earth, and is used at times as long as it is potable, and at times when not potable. With all this before us, should we at times be surprised on seeing bad concrete?"

Stanton (**10**) gave us a method for making expansion bars which indicate when there is unfavorable reaction of aggregates in concrete. He also gave us data on numerous combinations of aggregates and of different size gradation. It is of particular interest to note that any deviation resulted in changes in the expansion as measured by the usual bars. He coined the word "pessimum" and stated that there was probably "a different 'pessimum' amount of alkali for various minerals, just as there is a different 'pessimum' amount of different minerals for a given alkali content."

Using the word "pessimum" as an antonym to the word "optimum," there can be a "pessimum" condition either for the best results or another "pessimum" condition for the worst results. Any change in the chemical or physical condition in any critical concrete system may alter the "pessimum" values—variables such as chemical composition, fineness, temperature, moisture, any cycle change in storage, and design of concrete mix.

Any freshly mixed concrete is an active chemical mixture which must be studied as a whole and properly designed to obtain only beneficial results from the chemical reactions. In some cases we are learning correct procedure by observing the effects of faulty operations of the past. Our aim must be to properly evaluate any mixture before use and, in doing this, seemingly things of little importance may well determine the correct procedure.

When the investigation of cracked concrete in 1934 was made, to which I have already referred, my attention was called to a number of other structures in the same vicinity which were equally bad and where, in each case, a separate brand of cement was used, and in each case extra care had been given to the work. But I was fortunate enough to see a large structure which seemed to be in excellent condition although the kind, type, and proportion of aggregates were said to have been the same as in the first unfortunate building, with the single exception that still another brand of cement had been used.

In 1934 none of us was "alkali conscious" and probably nobody actually knows the alkali content of the different cements used on the different structures. From tests that have been made, however, I am reasonably certain that the total alkalies, calculated as Na_2O, must have been between 0.70 and 1.1 per cent approximately and thus only "high-alkali cement" was used on both the good and the reactive concrete. Why, then, did the one structure mentioned turn out well? My answer to this is that it received a coat of waterproofing.

As a consultant on another structure, in January of this year, I found that the concrete had been fabricated about 1927, using a cement of good repute, local sand now known to be reactive, and a coarse aggregate of good record from a distant source. Vertical walls of exposed concrete in one wing were so badly cracked that rebuilding had been recommended. There were two towers in this wing and each had two partial domes which were not cracked although constructed at the same time under the same conditions as the balance of the concrete in the wing. They had been painted on the outside with a waterproof paint. The two cases mentioned together with others that I have seen thoroughly satisfy me that concrete suspected of having a potentially unfavorable chemical reaction may, and should be protected from moisture.

Most of us have heard the desire expressed for some of the "good old fashioned cement" such as was used thirty years or more ago and some of us have made investigations to see what could be uncovered. It is difficult to make thorough investigations in any district and at best any investigation must be open to some question. However, I am satisfied that, in the very district which I have mentioned as having unfavorable chemical reaction of aggregates, the concretes could have been more satisfactory if they had used the "good old fashioned" unwashed aggregates and gravel from the local pits containing reactive aggregates. In fact I have seen at least one structure where apparently the only part free from cracks used all local reactive aggregates.

Even though some jobs were improved by the use of reactive aggregates I do not advocate their promiscuous use. The same jobs could also have been made satisfactory by the use of only good aggregate, and this would be the safer practice of the two. Sand and gravel pits with reactive aggregates may be of variable composition and there must be the "pessimum" amount of reactive particles to insure good work (also considering all other variables).

Still other districts may not have enough reactive material in the deposits to reach the "pessimum" amount for satisfactory concrete, even when using all of the aggregates. In some such cases the use of more of the fine material containing reactive particles is recommended. However, as the amount of reactive material added is critical, care must be taken to see that a sufficient quantity is added to get past that "pessimum" point where conditions are worst. In some cases the natural fines of the sand must be washed out as they contain too much organic or other undesirable material. I have, however, in some such cases recommended the addition of some of the ground material after washing (**6**).

Having more fines such as silt or rock dust is not original with the author and has been mentioned by others: Larmour (**9**), Davis (**3**), Stanton, Blanks, and Meissner (**2**) and Bates (**1**, p. 83). It is probable that in cases where the aggregates have reactive material such as opal, andesite, rhyolite, and other volcanic rocks in amounts and size gradation such that the most unfavorable reactions would occur in the concrete, a small addition of ground material, such as pozzolana high in opal, would adjust the mixture so that reactions would produce concrete without unfavorable expansion. Pozzolanas are not a cure-all for concrete troubles. This much, however, can be said that, based upon experience and the results of tests (others as well as our own), the replacement of cement with suitable pozzolanas in the correct amount and in the proper physical state produces concrete containing reactive aggregates, without unfavorable expansion. In some cases 30 to 40 per cent of the cement must be replaced with the fine active rock dust or pozzolana.

EVALUATION OF POZZOLANAS

Just calling a material a pozzolana does not indicate that it will be a suitable material for concrete in every way. A suitable pozzolana for use in regulating the chemical reactions of aggregates in concrete must be reactive with the solutions in the concrete and there must be sufficient of this pozzolana finely comminuted and well mixed so as to react with all of the solutions in the concrete. The material used must be such that the proper workability and strength in the concrete may be obtained. It is preferable to place the pozzolanic concrete than to pour it.

Because of the high specific surface of ground pozzolana, more water will be required for gaging the concrete in which it is used. Kalousek and Jumper (**8**) stated: "The portland pozzolana pastes were generally very sticky compared to those of portland cements. Also, even though the flows were comparatively low, in a number of cases the pozzolana cement mixes were markedly 'fatter' than those of the portland cement, permitting easy fabrication of specimens."

Failure to recognize the true characteristics of portland-pozzolana cement concrete and the use of too much water will result in poor quality. The cost must be about as low as for the usual cement concretes.

Where there is incompatability between types of aggregates or between the aggregates and the solutions in the concrete, the use of the proper pozzolana as a corrective will be an added benefit to concrete used in sulfate waters (**8**). The use of fine pozzolana will tend to give more shrinkage in the concrete but I have seen no objection to this.

In making concrete we must have all possible information before us and I know there are numerous combinations to be considered. The cure for trouble in one concrete may not apply in another. If the need of a pozzolana is indicated, it may be used in concrete without too much trouble and probably at no extra expense. Where pozzolanas are not needed, good concrete can be fabricated without them.

The evaluation of a pozzolana has not been developed to an A.S.T.M. standard or even to an A.S.T.M. tentative. Since methods now in use may either require too much time or may not be conclusive we must use discretion in the evaluation of pozzolanas.

The late Guy W. Jordan, Chief Chemist of the Southern States Portland Cement Co. gave us a method which he thought would be a measure of activity.[4] He also presented data. Quoting from Mr. Jordan:

"Various investigators have found that in Portland-pozzolan reactions the alkalies in the *cement* act as a catalyst. Some of the essential reactions may be as follows:

$$2NaOH + SiO_2 \rightarrow Na_2O \cdot SiO_2 + H_2O$$

$$Na_2O \cdot SiO_2 + Ca(OH)_2 \rightarrow CaO \cdot SiO_2 + 2NaOH$$

$$CaO \cdot Al_2O_3 \cdot 2SiO_2 + 2NaOH \rightarrow Na_2O \cdot CaO \cdot Al_2O_3 \cdot 2SiO_2 + H_2O$$

$$Na_2O \cdot CaO \cdot Al_2O_3 \cdot 2SiO_2 + Ca(OH)_2 = 2CaO \cdot Al_2O_3 \cdot 2SiO_2 + 2NaOH$$

"From this it follows that the extent of a reaction between a pozzolan and an alkali such as sodium hydroxide would also be a measure of its pozzolanic activity.

"The method for determining this activity is comparatively simple. To a 250-ml. Pyrex Extraction flask is attached a Friedrichs reflux condenser using a No. 8 one-hole stopper and the proper hose connection for the water inlet and outlet. In making the test exactly 1 g. of the pozzolanic material, ground to pass a No. 100 screen, is placed in the extraction flask and exactly 100 ml. of N sodium hydroxide is added. The flask is then suitably heated until reflux condensing starts and is continued for one half hour. Filter through a 11-cm. folded filter paper, washing several times. Discard the residue, add 25 ml. of HCl (1:1) to the filtrate, evaporate to dryness and bake until the silica is dehydrated. Take up with a little HCl (1:1) being sure to have enough volume so that all the NaCl will be in solution. Filter, wash free of chlorides, and determine the silica in the customary manner. Heat the filtrate, add NH$_4$OH until the R$_2$O$_3$ is precipitated, filter and determine R$_2$O$_3$ by the customary procedure. The sum of the silica and the R$_2$O$_3$ will be a measure of the pozzolanic activity of the particular material being tested.

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[4] Letter dated June 18, 1946, to Sponsoring Committee on Blended Cement of A.S.T.M. Committee C-1 on Cement.

"Tested by the above procedure, the following results were obtained on a number of pozzolans submitted:

	Pozzolanic Activity
1. Diatomaceous earth—calcined	43.85
2. Diatomaceous earth—raw	33.35
3. Monterey shale—calcined	68.85
4. Monterey shale—raw	43.50
5. Pumicite—calcined	42.90
6. Pumicite—raw	29.50
7. Tuff—calcined	42.30
8. Tuff—raw	44.50
9. Granite A—calcined	6.15
10. Granite A—raw	7.35
11. Basaltic tuff—calcined	16.10
12. Basaltic tuff—raw	15.15
13. Clay A—calcined	21.25
14. Clay A—raw	7.90
15. Chicago Slag (P3) ground	8.65
16. Chicago fly ash (P8)	15.35
17. Pacific Portland quenched (P60)	12.50
18. Pacific Portland composite sample (P62)	12.00

"Since the first essential of a good pozzolanic material is its ability to react (combine) with alkalies, it follows that certain materials which liberate rather than combine with alkalies, such as a volcanic ash of the leucite type, are far from being desirable pozzolanic materials. And since the reactivity which determines the concrete strength of a portland cement-pozzolan mixture is partly due to the alkali catalyst, coming from the portland cement, it also follows that the concrete strength test is a measure of the activity of the portland cement-pozzolan mixture but not of the pozzolan as a separate material.

"In our test just described for pozzolanic activity, we have found very poor correlation with concrete strengths except that in general the higher the alkali content of the cement, the higher the concrete strengths of the portland-pozzolan mixture. There is however a very good correlation between this test for pozzolanic activity and the sulfate resistance of 1:4 mortar bars. For instance we found that a minimum of twenty per cent of reactive pozzolanic material was necessary in a portland-pozzolanic mixture to obtain a satisfactory sulfate resistant cement and the higher the activity of the pozzolan the less of it was required in the mixture.

"While we have not made any extensive tests in our laboratory for the purpose of using pozzolan to prevent expansion due to alkali-aggregate reaction, our conversations with others would lead us to believe that there is a very good correlation between this test for pozzolanic activity and the ability of the pozzolan tested to prevent expansion due to alkali-aggregate reaction.

"It is our personal belief that tests for sulfate resistance or tests for prevention of expansion due to alkali-aggregate reaction would give you a much better correlation with any test for pozzolanic activity which may be selected, as compared with concrete strengths."

Stanton's (10) bar test in moist sealed containers is possibly the best method that has been proposed for cement-aggregate combinations and the fine pozzolana to be tested would be a part of any combination selected for test.

TABLE II. —MODIFIED CEMENT, SAMPLE NO. 24157 —CHEMICAL ANALYSIS.

Constituent	Per Cent
SiO$_2$	20.98
Al$_2$O$_3$	5.09
Fe$_2$O$_3$	4.10
CaO	62.34
MgO	4.42
SO$_3$	1.72
Loss on ignition	0.86
Total	99.51
C$_3$S	49.30
C$_2$S	23.00
C$_3$A	6.60
C$_4$AF	12.50

TABLE III.—MODIFIED CEMENT *VERSUS* PORTLAND-POZZOLAN—PHYSICAL CHARACTERISTICS.

		Sample No. 24157		Sample No. 24158	
Bin		XXIX-180		81.8% C. + 18.2% Shale	
Type		Modified M.W.D. No. 79		Portland Pozzolan	
Fineness (passing No. 200 sieve), per cent		95.4		96.8	
Initial set		2 hr. 35 min.		3 hr. 57 min.	
Final set		5 hr.		5 hr. 25 min.	
Soundness—steam		O.K.		O.K.	
Normal consistency, per cent		22.8		28	
Water in standard mortar, per cent		10.3		11.2	
		Tensile	Compressive[a]	Tensile	Compressive[a]
Strength Tests, psi.:					
Standard	1 day	173	902	155	782
	3 days	248	1803	276	1856
	7 days	318	2839	371	2904
	28 days	408	4149	505	5117
	3 months	434	4405	576	6184
	6 months	402	5166	597	6700
	1 yr.	395	4547	642	7225
	2 yr.	355	5107	625	6474
	5 yr.	347	4913	621	7957
	10 yr.	302	4329	679	6133
	3 months air	543	5269	569	6227
	6 months air	515	5365	504	6279
	1 yr. air	474	5185	476	6545
	2 yr. air	401	5275	433	6213
	5 yr. air	748	6514	459	7013
	10 yr. air	883	6960	768	8021
1 part cement 3 parts limestone	1 day	197	951	142	781
	3 days	377	2324	318	1941
	7 days	538	3711	484	3463
	28 days	656	4748	696	5205
	3 months	760	5515	744	6017
	6 months	809	5669	782	6207
	1 yr.	783	6221	754	6791
	2 yr.	821	6207	803	6697
	5 yr.	853	6483	819	7362
	10 yr.	869	6080	833	7576
	3 months air	762	5877	705	6582
	6 months air	753	5460	728	6532
	1 yr. air	558	5363	648	5921
	2 yr. air	499	5340	579	6348
	5 yr. air	724	5714	859	7115
	10 yr. air	1020	7300	1004	7894
1 part cement 1 part Colton plaster sand 2 parts limestone	1 day	192	918	137	644
	3 days	372	2179	300	1794
	7 days	518	3571	443	3231
	28 days	649	4570	649	5035
	3 months	695	4893	793	6163
	6 months	753	6029	815	6837
	1 yr.	810	5966	704	6505
	2 yr.	814	5834	777	8096
	5 yr.	806	6833	797	7427
	10 yr.	770	6960	751	7491
	3 months air	749	6243	872	6514
	6 months air	714	5917	779	6792
	1 yr. air	582	5699	609	6685
	2 yr. air	611	5564	639	6793
	5 yr. air	867	7088	900	7268
	10 yr. air	1109	7686	1065	7978

[a] All compression tests were made on 2 by 4-in. cylinders.

NOTE.—Unless mentioned to the contrary, the sand used was standard Ottawa sand, 20–30.

All strength specimens were stored in the damp closet 1 day at about 70 F. before they were removed from the molds. They were then stored in water at 70 F.

Those specimens marked "air" were stored in water until 28 days of age and then they were stored in air in the attic where they were subject to the changes in temperature and moisture of the seasons but not directly exposed to moisture.

All specimens were molded by the same man, an experienced and dependable operator. All results reported are, with a single exception, the averages of three specimens.

It is of special note that the strengths of the specimens with the pozzolan admixture were, on an average, 14 per cent higher than the portland cement used by itself.

Stanton's bar test may also well be used to indicate a possible source of pozzolana. The proper amount of coarse-sized material could be tested in a neutral sand-cement mixture. If a reactive aggregate is not indicated then the material would not be a good pozzolana when finely pulverized. Another way would be to take a destructive combination and add fine materials, suspected to be pozzolanas, in different amounts. A good pozzolana will stop the destructive reaction.

TABLE IV.—SIEVE ANALYSES OF COLTON PLASTER SAND AND LIMESTONE.

Sieve Size	Percentages Passing Each Sieve	
	Plaster Sand	Limestone[a]
No. 3	100	100
No. 4	99.9	99.93
No. 8	89.8	92.98
No. 14	69.1	73.68
No. 30	41.2	50.53
No. 48	17.1	34.13
No. 100	4.15	21.09
No. 200	1.85	14.13

[a] The Colton limestone was clean material crushed and screened.

Paragraph 243 of Detailed Requirements, Section II of Standard Government Form of Invitation for Bids No. 694-35-9, under date of May 11, 1935, gives a "Test for activity with hydrated lime."[5] I have used this test and obtained very high results with active pozzolana.

Strength tests of different kinds have been used but these alone apparently do not indicate which pozzolanas are active in the correction of "clashing" aggregate combinations. The best pozzolana that I have seen in commercial amounts is a shale, which we call "Phillips Shale," high in opal, from the Puente Formation, also called by some, Monterey Formation, in Los Angeles County, Calif. The shale is calcined at about 1500 F., and then pulverized. We made a series of tests using portland cement and another series using this same cement plus 18.2 per cent of the above calcined shale or pozzolan. A report on this series is given in Tables II, III, and IV.

In making the tests with the cement-pozzolan mixture, 1.1 parts of the mixture was used instead of 1 part of cement. In other words the pozzolan admixture was treated more as an admixture rather than as a substitute for cement.

TABLE V.—ANALYSES OF CALCINED PHILLIPS' SHALE.

	Dec., 1938	May, 1947
SiO_2	68.52	64.94
Al_2O_3	11.24	12.90
Fe_2O_3	4.14	5.44
CaO	5.15	6.20
MgO	2.32	3.32
SO_3	0.34	0.31
Loss on ignition	4.70	4.01
Na_2O	1.13	1.14
K_2O	1.41	1.58
	98.95	99.84
Total alkalies as Na_2O	2.06	2.18

DISCUSSION OF RESULTS

The results of strength tests on specimens stored in air are of particular interest, since the general feeling of most people I have talked with has been that the strength would be lower than with portland cement only.

The calcined shale seems to be remarkably uniform over a period of years and two analyses of average samples are given in Table V.

When this active calcined shale or pozzolan (about 90 per cent passing No. 325 sieve) is added to a reactive aggregate-cement combination, or as a substitute for cement, the expansion, as determined by bars in the Stanton sealed container method, may be brought to less than 0.1 per cent after years of storage (Figs. 7 and 8). In the occasional cases where reactive aggregates must be used, it is recommended that

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[5] U. S. Engineers Office, Second Portland District, 627 Pittock Block, Portland, Ore.

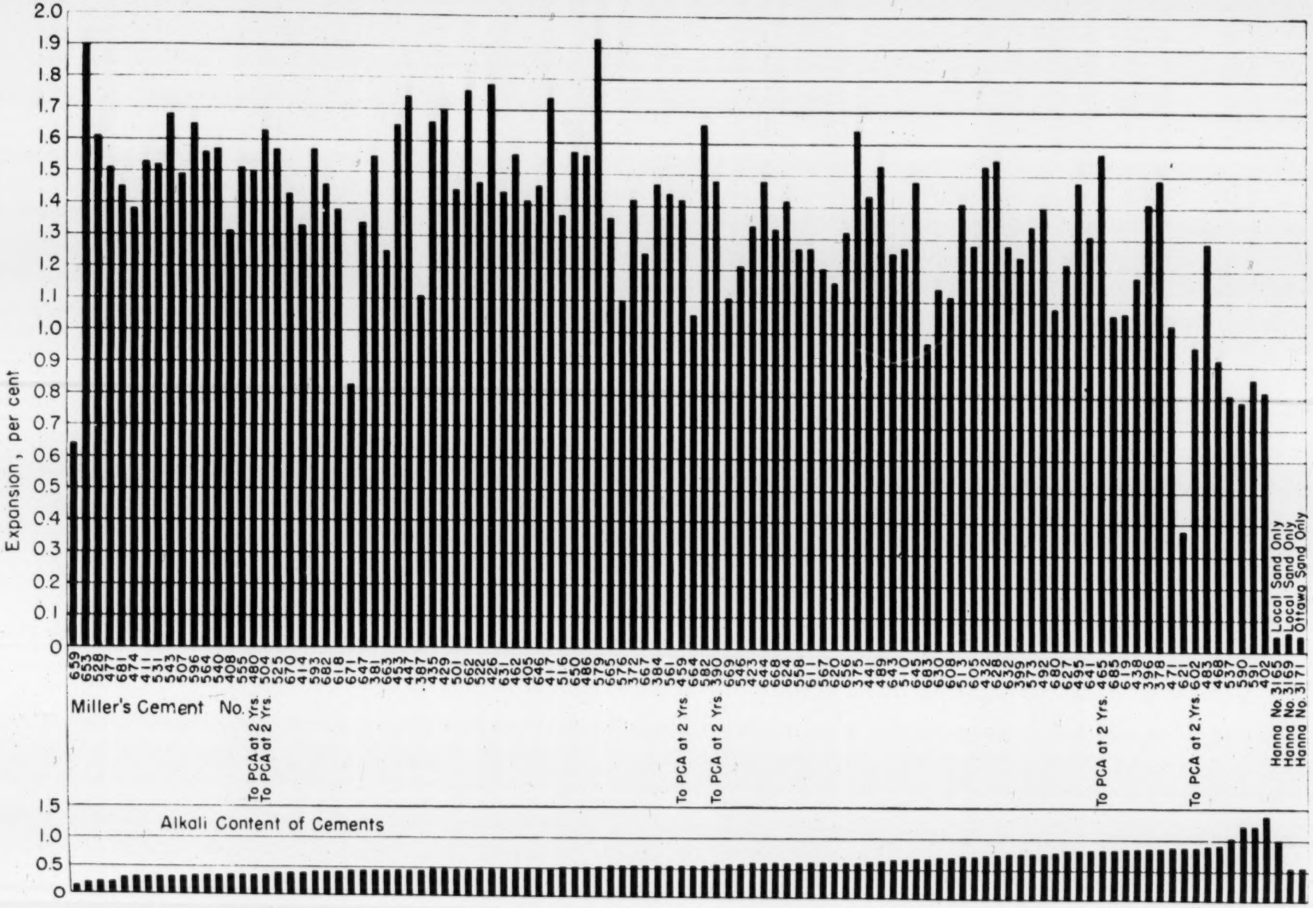

FIG. 5.—Expansion of Bars in Closed Containers at Age of Four Years. Alkali content of cement progressing from lowest at left. Extreme right has three cements without reactive aggregate.

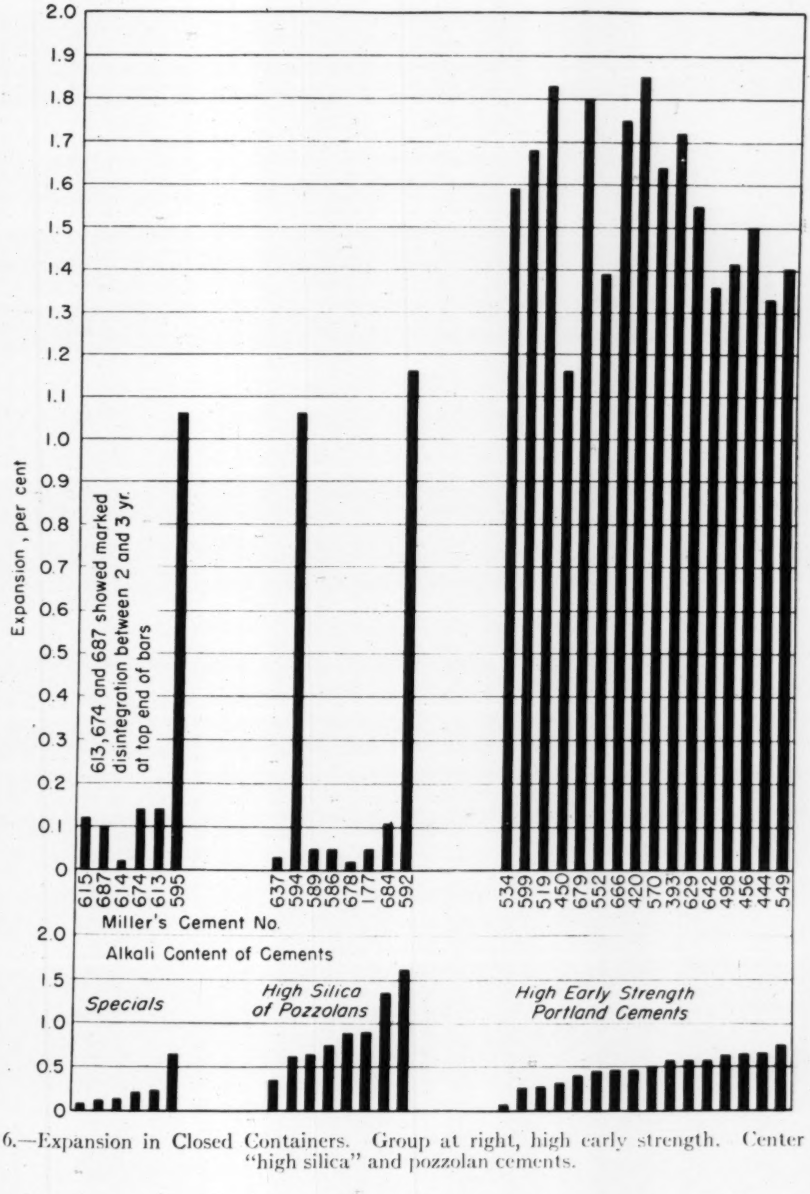

FIG. 6.—Expansion in Closed Containers. Group at right, high early strength. Center group, "high silica" and pozzolan cements.

consideration be given to the use of the proper amount of such a pozzolana as a part of the aggregate.

In 1943 the author (6) gave a discussion and showed results of tests on numerous cements up to the age of 18 months. Figures 5 and 6 show these results at the age of four years with the cements arranged so that the lowest alkali is on the left and increasing to the right.

It is to be noticed that the total alkali

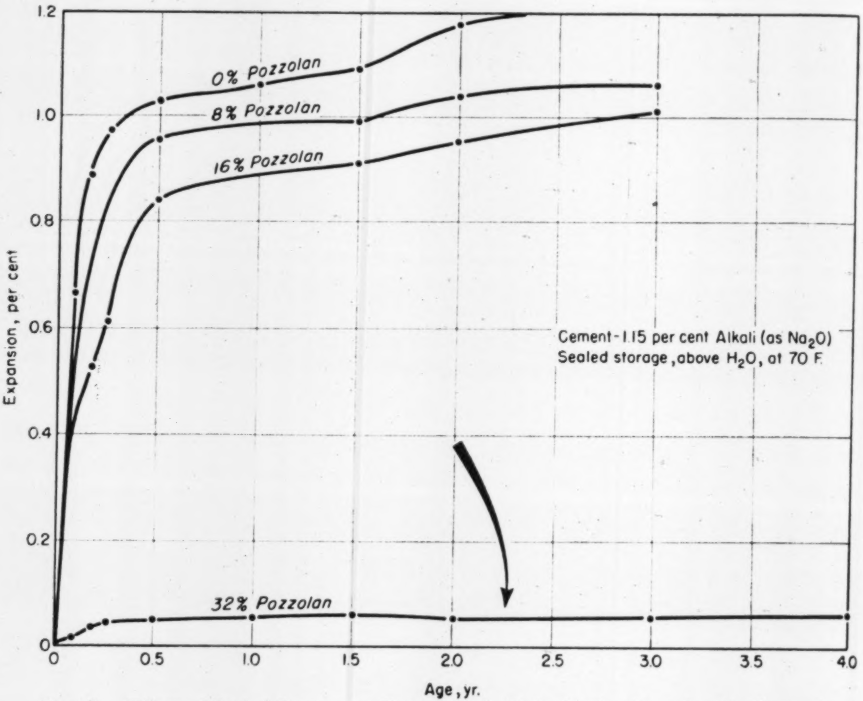

FIG. 7.—Effect of Sealed Storage on Expansion by Substituting Pozzolan for Cement.

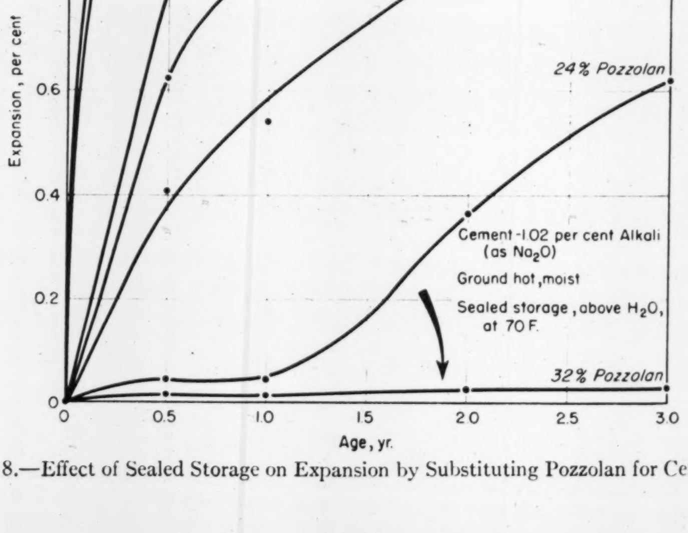

FIG. 8.—Effect of Sealed Storage on Expansion by Substituting Pozzolan for Cement.

content of these portland cements varied from less than 0.2 per cent to approximately 1.4 per cent and the expansion was high in every case for both regular portland cements and high early strength portland cements. At the extreme right of Fig. 5, tests on three samples of cement without the bad aggregate are given, two using only the "neutral" aggregate that was used in the combination with the bad aggregate and one where only Ottawa sand was used. It will be noticed that the expansion of each of these three samples is very low.

Figure 6 shows the so-called high-silica or pozzolan group and it will be observed that several of these after four years still have less than 0.1 per cent expansion. This confirms my previous statements that a suitable pozzolana will properly neutralize an unfavorable cement-aggregate combination.

Figures 7 and 8 very definitely show the pronounced beneficial effect of this pozzolana in controlling expansion as measured by the Stanton bar test in moist sealed containers.

CONCLUSION

The conclusions from this work are that the use of the correct pozzolana is indicated in the occasional cases when reactive aggregates must be used, and its use in concrete is recommended to control reactive aggregates. When aggregates are not harmfully reactive the use of pozzolana in concrete is not necessary.

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(7) George L. Kalousek, "Studies of Portions of the Quaternary System Soda-Lime-Silica-Water at 25 C.," *Journal of Research*, Nat. Bureau Standards, Vol. 32, No. 6, June, 1944, p. 301.

(8) George L. Kalousek and C. H. Jumper, "Some Properties of Portland Pozzolana Cements," *Proceedings*, Am. Concrete Inst. Vol. 40, pp. 145 and 161 (1943).

(9) H. McC. Larmour, "Proceedings of Conferences for Discussion of Problems Related to Alkalies in Cement and Their Effect on Aggregates and Concretes," Bureau of Reclamation, Denver, 1941, p. 82.

(10) Thomas E. Stanton, "Expansion of Concrete Through Reaction Between Cement and Aggregate," (without discussion) *Proceedings*, Am. Soc. Civil Engrs., December, 1940, p. 1781; also (with discussion) *Transactions*, Vol. 107, p. 54 (1942).

(11) Jan Struther, "A Pocketful of Pebbles," Harcourt, Brace, New York, N. Y.

(12) Bailey Tremper, "The Effect of Alkalies in Portland Cement on Durability of Concrete," *Journal*, Am. Concrete Inst., November, 1944; *Proceedings*, Am. Concrete Inst., Vol. 41, p. 91 (1941).

DISCUSSION

Mr. A. D. Conrow[1] *(presented in written form)*.—Mr. Hanna has presented some information on a subject that is of much interest to all engaged in the manufacture and use of portland cement.

During 1936 and 1937 when serious cracking of concretes containing the sand gravel aggregates of Kansas and Nebraska began to attract notice, the writer started some research to try to learn the cause and to develop a remedy if possible. One of the requisites for studying a disease is to be able to develop the disease. In 1938, somewhat by accident, we learned how to develop the disease and quite extensive exploratory tests were made in attempts to control or prevent it. Our facilities were not adequate to search deeply into its causes, and our efforts were directed mainly in efforts to prevent or inhibit the disease by what may be termed "cut-and-try" methods. In the course of these efforts we found that there is a great similarity in our findings when dealing with the reactive sand gravel aggregates of Kansas and Nebraska to those found by Mr. Hanna with California reactive aggregates. We have also found that if a cement composed of a blend of cement and an active pozzolana is used in concrete made with the reactive aggregate little evidence of the disease develops.

We have further found that no matter how low the content of sodium and potassium alkalies may be in a portland cement, there will be potential conditions present for the development of destructive expansion if a reactive aggregate is used, for we have developed it with a cement in which the sodium and potassium alkalies in terms of the sodium equivalent were as low as 0.09 per cent and its development was greater than for many cements in which the alkali content was as high as 0.7 per cent.

This indicates to us that the predominent reactive ingredient contributed to concrete by the cement is something other than the sodium and potassium alkalies and that possibly these very active alkalies act as either catalysts or accelerators of the action of the predominating reactant. If the presence of the predominating reactant is prevented, or when present it is neutralized, absorbed or converted to a harmless state, a durable concrete will be obtained with the aggregates in question.

Reflection suggests that this predominating reactant may be the calcium hydroxide liberated when portland cement is hydrated.

Some recent experiments in our laboratory indicate that portland cement paste hydrated at 100 F. for a period of 7 days contains as high as 20 per cent of calcium hydroxide, and more is liberated with longer periods of hydration. This indicates the paste at this stage and for a quite considerable period thereafter contains a high percentage of a very active constituent. It is logical to believe that if this constituent remains in the active form for a long period undesirable reactions may occur unless

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[1] Research Engineer, Ash Grove Lime and Portland Cement Co., Chanute, Kans.

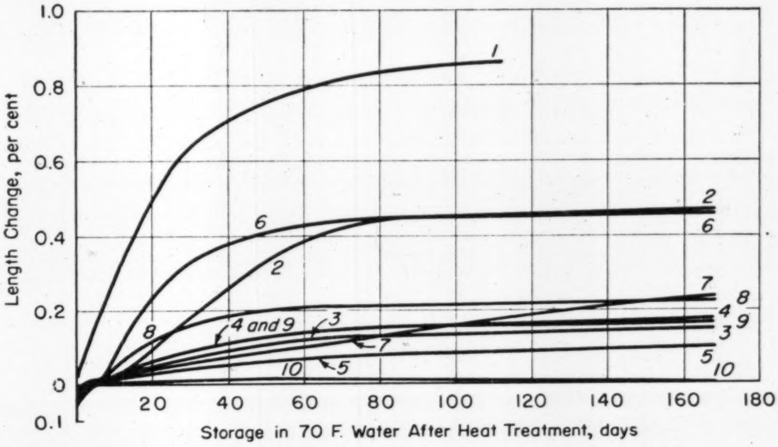

FIG. 9.—Expansion of Concrete Made with Reactive Aggregate.

Concrete exposures previous to the above treatment

1. Curing in water at 70 F., varying 50 to 130 days
2. Temperatures raised in water to 160 F., varying 10 to 14 days
3. Dried in drying oven at 160 F., varying 10 to 30 days
4. To above storage

Curve 1—Standard portland cement, alkali 0.4 per cent
Curve 2—Standard portland cement, alkali 0.6 per cent
Curve 3—Pozzolanic blend with portland cement, 10 per cent pozzolana substitution
Curve 4—Pozzolanic blend with portland cement, 20 per cent pozzolana substitution
Curve 5—Pozzolanic blend with portland cement, 30 per cent pozzolana substitution
Curve 6—Commercial portland pozzolan cement
Curve 7—Commercial portland pozzolan cement
Curve 8—Commercial portland pozzolan cement
Curve 9—Commercial portland pozzolan cement
Curve 10—Commercial portland pozzolan cement

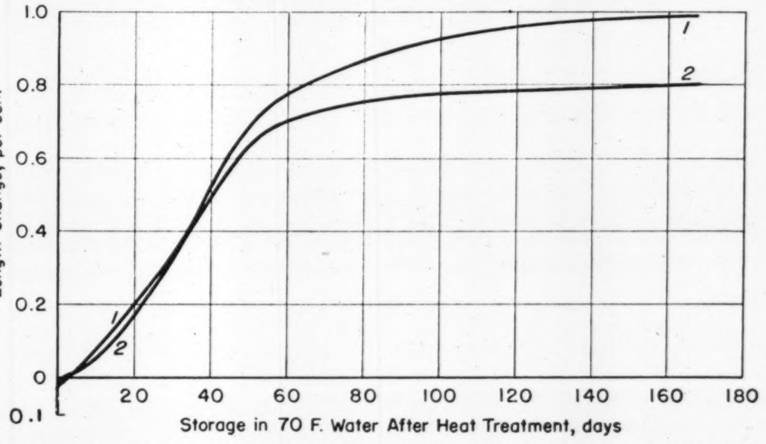

FIG. 10.—Expansion of Concrete Made with Reactive Aggregate and Very Low Alkali Content

Concrete exposures previous to the above treatment

1. Curing in water at 70 F., 117 and 56 days
2. Temperatures raised in water to 160 F., 11 and 8 days
3. Dried in drying oven at 160 F., 45 and 29 days
4. To above storage

Curve 1—Cement having 0.03 per cent alkali
Curve 2—Cement having 0.09 per cent alkali

conditions are such that the active constituent is converted to a stable state either by accident or design, early in the life of the concrete.

If the predominant reactant is calcium hydroxide, a pozzolanic material is the most logical material to use to convert it to a stable state and the more active the pozzolana, the better. Lime pozzolana blends are good cements in themselves, though slow hardening, and contribute their strength to the blend with the result that the ultimate strength of the concrete may be as great when made with the blended cement as it would be if standard portland cement had been used.

The accompanying Figs. 9 and 10 show the expansions obtained from some of our tests.

Figure 9 shows the expansions of concretes made with and without pozzolanas. It may be seen that all commercial portland pozzolanas are not effective—note No. 6.

Figure 10 shows expansions obtained on concretes made with cements containing very low alkali.

The exposure of these concretes previous to development of abnormal expansion may be subject to comment. This exposure is given on the lower part of Fig. 10. This exposure included the following:

1. A considerable curing period in water at 70 F.
2. A considerable period in a water bath at temperature up to 160 F.
3. A considerable period drying in a drying oven at temperatures up to 160 F.

It may be said that the 160 F. temperature is excessive and not likely in nature. This is true. Our experiments indicate the cement paste must come to a certain degree of hydration before abnormal expansion develops. It may take a very long time for it to reach that degree at 70 F. Application of heat and moisture will hasten hydration to the degree necessary for development of abnormal expansion and is necessary if information is to be obtained quickly.

The writer has found that exposure to the heat treatments, saturated and dry, at 130 F. causes as great expansions as that caused by the 160 F. exposure, and that temperature is now being used with results much the same as has been presented in Figs. 9 and 10.

Finally, low alkali cement does not appear to be a comprehensive solution to the problem. Judicious blends of portland cement and an active pozzolana show good promise of an improvement of concrete in which a reactive aggregate is used.

MR. L. P. WITTE[2] (*presented in written form*).—The following test method is presented because it appears to have potentialities as a simple means for evaluating the effectiveness of reactive siliceous materials in reducing the alkali-aggregate expansion described in Mr. Hanna's paper. This test consists mainly of determining the reduction in expansion resulting from the replacement of cement by the siliceous material in a standard mixture of crushed pyrex glass and high-alkali cement. Pyrex glass with the minus 100 size removed is very reactive and causes considerable expansion when mixed with high-alkali cement. The minus 100-size glass apparently has the same effect in reducing expansion as other finely divided reactive siliceous materials. Pyrex glass was chosen for this test because its composition is less apt to vary. A description of the test follows:

The control or reference mix is comprised of 1.00 part modified high-alkali cement (sodium equivalent 1.23 per cent), 2.25 parts of pyrex glass (graded

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[2] Materials Engineer, U. S. Bureau of Reclamation, Denver, Colo.

20 per cent each of No. 8 to No. 4, 16 to 8, 30 to 16, 50 to 30, 100 to 50 sieve sizes) and a water-cement ratio of 0.50. After mixing, three 1 by 1 by 10-in. bars are fabricated and moist cured for 24 hr. Mixing and fabrication procedure is very similar to A.S.T.M. Standard Method of Test for Autoclave Expansion of Portland Cement (C 151 – 43)[3]. Initial length measurements are taken at the end of the 24-hr. curing period and the bars placed in sealed storage containers with a small amount of water and stored at constant temperature of 100 ± 3 F. Water in the container does not come in contact with the specimens. Length measurements are taken at weekly intervals during the first month and monthly thereafter.

TABLE VI.—REDUCTION OF ALKALI EXPANSION BY POZZOLANA AND PULVERIZED GLASS REPLACEMENTS.

100 per cent pyrex glass—graded 20 per cent each of Nos. 8, 16, 30, 50, and 100 sieve sizes.
Mix 1:2.25.
1 by 1 by 10-in. mortar bars.
Cement No. 3562 (1.23 per cent alkali).
Sealed moist storage at 100 F.

Test Material	Test Material Replacing Cement by Weight, per cent	Reduction of Expansion at 14-days Age, per cent
Pozzolana P-129 Phillips Shale calcined 1500 F	10	64
	20	88
	50	100
Pozzolana P-180 Monolith Tufa Rock calcined 1400 F	10	49
	20	74
	50	100
Pulverized pyrex glass[a]	10	65
	20	92
	50	105

[a] Glass ground to 99 per cent passing No. 325 sieve.

The mix containing the replacement is made identical to the reference mix except that varying percentages of the cement are replaced with test material.

Reduction of expansive reaction is expressed as a percentage and is calculated by the formula

$$R_e = \frac{E_r - E_t}{E_r} \times 100.$$

where

R_e = percentage reduction of expansion reaction,

E_t = average expansion of three bars fabricated from the test mix, and

E_r = average expansion of three bars fabricated from the reference mix.

The reduction in alkali expansion brought about by 10, 20, and 50 per cent (by weight) replacements of cement with two different pozzolanas and pulverized pyrex glass is shown in the accompanying Table VI. It is recognized that the large reductions shown in the table are not due entirely to the test materials. Part of the reduction is due to the lowering of the cement in relation to the aggregate (glass). However, the total reduction is real and the test appears to offer a simple means for testing the effectiveness of materials in reducing alkali-aggregate expansion. Pulverized pyrex glass was included in the table primarily to show the possibilities of using this type of test for determining the reactivity of aggregates.

MR. R. E. DAVIS.[4]—Mr. Hanna's paper holds a good deal of interest for me. For the past several years I have been advocating the use of pozzolanic materials for certain types of concrete construction, but it was not always thus. Back in 1928, when it was suggested to me that a pozzolanic material be used in dam construction in California, I recommended against its use because results of laboratory tests generally indicated a greater drying shrinkage in concrete where pozzolans were employed.

In later years, as I traveled about the country examining concrete, I had opportunity to compare dams and other structures containing straight portland cement and washed aggregates with those in which there had been employed the old sand cements, pozzolana cements, large quantities of stone dust, and what were then regarded as excess fines in the sand. What I saw convinced me of the benefits to be derived in mass concrete construction, particularly dams, through the use of a considerable percentage of finely divided siliceous material. For comparable conditions, those structures which contained such materials on the average exhibited substantially less tendency toward cracking and a much greater degree of watertightness.

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[3] 1946 Book of A.S.T.M. Standards, Part II, p. 14.

[4] Professor of Civil Engineering and Director, Engineering Materials Lab., University of California, Berkeley, Calif.

The results of investigations of blended cements, begun at the University of California in 1933, demonstrated that in the presence of moisture at normal temperatures all finely divided siliceous materials are reactive with lime. The rate of reaction is slowest for the coarsely crystalline silicas, such as quartz, is much more rapid for materials high in glass, such as pumicites and fly ashes, and is most rapid for materials high in opal.

The generally superior condition of structures containing finely divided siliceous materials capable of combining with the lime liberated during the process of hydration of the cement led me to the conclusion that the use of a suitably active pozzolana was desirable in all heavy concrete construction.

It has become general knowledge that much of the severe cracking observed in concrete structures in a number of regions of the United States is caused primarily by the reaction between certain siliceous aggregates and the alkali contained in the cement. In all cases, the reactive minerals fall into the general class of pozzolanic materials; that is, in a finely divided state, they are all capable of quite rapid reaction with lime. In no case does excessive expansion occur in concrete when the reactive minerals are finely ground. This was our first clue to a possible means of correcting excessive expansion through the use of a finely divided pozzolanic material, preferably high in opaline silica, either as an addition to, or as a replacement of a portion of, the cement. Mr. Hanna's investigations have clearly demonstrated the effectiveness of such a procedure.

Not all the ills of a concrete can be charged to the ingredients which it contained at the time of mixing. Destructive compounds often find their way into the concrete from the surrounding medium. The effect of alkaline ground waters is well known. To combat the attack of such waters we have our sulfate-resistant portland cements, which are greatly superior to cements high in tricalcium aluminate. Not so well known, but a fact which long-time tests seem to have proven conclusively, is that the resistance of a concrete to sulfate action, regardless of the composition of the portland cement which it contains, may be substantially improved by the use of suitable amounts of finely divided pozzolanas, particularly those high in opal.

Recently there has been called to my attention the large growth and consequent cracking that has taken place in one of our dry docks that was built only five years ago. It is reported to me that this growth is in the order of 10 in. in a length of 1100 ft. There is no evidence that reactive aggregate was responsible for the growth, but there is evidence that the sulfates present in the sea water reacted with the lime that was liberated during the process of hydration of the cement, and also with alumina, to form calcium sulfo-aluminate crystals, which were responsible for a large part, if not all, of the growth. Here again, it seems probable that the use of a suitable pozzolana would have prevented excessive expansion.

At the beginning of this discussion, I stated that generally the drying shrinkage of concretes containing pozzolans were somewhat greater than for corresponding concretes containing straight portland cement. This is true for all cements, except type IV for which the drying shrinkage in concrete is about the same as for the usual portland-pozzolan cement employing either type I or type II as the portland-cement constituent. This larger drying shrinkage, however, is not a matter of importance in thick structures or in those not subjected to prolonged drying conditions.

In one other respect, concretes containing finely divided siliceous materials, including pozzolanas and the old sand cements, have exhibited inferior performance. Under severe conditions of freezing and thawing, their disintegration has been more rapid. It now seems probable that this was due to a smaller amount of naturally entrained air than that contained in corresponding concretes using straight portland cement. Results of recent investigations have demonstrated that, for equal amounts of entrained air, concretes using certain pozzolanas offer a resistance to freezing and thawing which is markedly superior to that of corresponding concretes using straight portland cement.

MR. WILLIAM LERCH[5] (*presented in written form*).—Mr. Hanna is to be commended for giving us the benefit of his extensive studies and observations on the chemical reactions of aggregates in concrete. Studies of this problem have been under way at the Research Laboratories of the Portland Cement Assn. since 1939. These studies have included a large number of laboratory tests of mortar prisms and tests of concrete prisms, beams, and slabs subjected to outdoor exposure at the Naperville, Ill. experimental farm.

Like Mr. Hanna, the Portland Cement Assn. has also directed the studies toward methods of preventing the alkali-aggregate reaction, or preventing the abnormal expansion and pattern cracking usually associated with the reaction. Part of this work included a study of the effect of powdered admixtures, by replacing part of the cement or part of the sand with the admixtures. Since Mr. Hanna has described the excellent results obtained with certain pozzolana materials, the following discussion will be confined to a description of the results the association has obtained with pozzolana admixtures.

Concrete Slabs:

In the first series of tests the effect of powdered admixtures in concrete was studied in a number of concrete slabs prepared for outdoor storage. One 30 by 42 by 6-in. slab for each condition of test was cast on the ground at the Naperville, Ill. experimental farm. The concrete mix was 1:2:3.2 by weight using Plainfield, Ill. sand and gravel as the neutral aggregate, and 95 per cent Plainfield sand plus 5 per cent siliceous magnesian limestone with Plainfield gravel as the reactive aggregate.

Six different cements and two admixtures were used. The cements were selected to have total alkali contents ranging from a high of 0.92 to a low of 0.17 per cent, calculated as Na_2O. The two admixtures were calcined shales. When the admixtures were used they replaced 15 per cent by weight of the cement.

The essential characteristics of the

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[5] Manager of Applied Research, Portland Cement Assn., Chicago, Ill.

TABLE VII.—CEMENTS, AGGREGATES AND ADMIXTURES USED IN 30 BY 42 BY 6-IN. CONCRETE SLABS AT THE NAPERVILLE, ILL., EXPERIMENTAL FARM.

(Series 285-6)

Mix 1:2:3.2 by weight using either Plainfield, Ill. sand or 95 per cent Plainfield sand plus 5 per cent siliceous magnesian limestone and Plainfield gravel.

Sample	Cement		Admixture Replacing 15 per cent of Cement	Siliceous Magnesian Limestone, per cent	Condition as to Pattern Cracking at 5 yr.
	Total Alkali as Na_2O, per cent	SO_3, per cent			
No. 5	0.92	1.73	None	5	Pronounced
No. 6	0.92	3.5	None	5	Pronounced
No. 7	0.92	3.5	Calcined	0	None
No. 8	0.92	3.5	Monterey Shale	5	None
No. 9	0.92	3.5	Calcined Shale from California	0	None
No. 10	0.92	3.5		5	None
No. 15	0.70	1.86	None	5	None
No. 16	0.70	3.5	None	5	None
No. 17	0.70	3.5	Calcined	0	None
No. 18	0.70	3.5	Monterey Shale	5	None
No. 1	0.55	1.55	None	5	None
No. 2	0.55	3.5	None	5	None
No. 3	0.55	3.5	Calcined	0	None
No. 4	0.55	3.5	Monterey Shale	5	None
No. 11	0.53	1.71	None	5	None
No. 12	0.53	3.5	None	5	None
No. 13	0.53	3.5	Calcined	0	None
No. 14	0.53	3.5	Monterey Shale	5	None
No. 20	0.34	1.87	None	5	None
No. 19	0.17	1.62	None	5	None

TABLE VIII.—CEMENTS, AGGREGATES AND ADMIXTURES USED IN 6 BY 6 BY 30-IN. CONCRETE BEAMS AT THE NAPERVILLE, ILL. EXPERIMENTAL FARM.

(Series 285-6)

Mix: 1:2:3.2 by weight calculating the 15 per cent of admixture a part of the sand. Aggregate: Elgin sand, or 95 per cent Elgin sand plus 5 per cent siliceous magnesian limestone and Elgin gravel.

Sample	Total Alkalies as Na_2O, per cent	Siliceous Magnesian Limestone, per cent	Admixture, 15 per cent	Condition as to Pattern Cracking at 4 yr.[a]	
				Beam A	Beam B
No. 31	1.05	0	Quartz	None	None
No. 32	1.05	5	Quartz	Pronounced	Pronounced
No. 33	1.05	5	Calcined Monterey Shale	None	None
No. 34	1.05	5	Fresno Pumicite	None	Pronounced
No. 35	1.05	5	Kansas Pumicite	Developing	Pronounced
No. 23	0.92	0	Quartz	None	None
No. 24	0.92	5	Quartz	Pronounced	Pronounced
No. 25	0.92	0	Calcined Monterey Shale	None	None
No. 26	0.92	5	Calcined Monterey Shale	None	None
No. 27	0.92	5	Fresno Pumicite	None	None
No. 28	0.92	5	Kansas Pumicite	Developing	None
No. 29	0.92	5	Calcined Mowry Shale	Pronounced	Developing
No. 30	0.92	5	Calcined Clay Lime	None	None
No. 36	0.68	0	Quartz	None	None
No. 37	0.68	5	Quartz	None	None
No. 38	0.68	5	Kansas Pumicite	Pronounced	Pronounced
No. 39	0.53	0	Quartz	None	None
No. 40	0.53	5	Quartz	None	None
No. 41	0.53	5	Kansas Pumicite	Pronounced	Pronounced
No. 42	0.21	0	Quartz	None	None
No. 43	0.21	5	Quartz	None	None
No. 44	0.21	5	Kansas Pumicite	None	None

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[a] A beams laid on the ground. B beams set upright in the soil so about half the length was exposed to weather.

composition of these slabs are shown in the accompanying Table VII. Observations of the appearance of pattern cracking after 5 yr. of outdoor exposure are also shown in the table.

Only two of the slabs, Nos. 5 and 6, show serious pattern cracking within five years. These slabs were made with the cement of highest alkali content, 0.92 per cent as Na_2O, without the calcined shale admixtures and with 5 per cent of the reactive siliceous magnesian limestone. The cement was used with two different SO_3 contents. These two slabs showed some cracks within a few months and they showed pronounced pattern cracking within one year. The higher SO_3 content delayed the cracking somewhat but did not prevent it.

When this same cement was used with 15 per cent of either of the calcined shale admixtures replacing cement, there was no evidence of pattern cracking with the reactive aggregate even after 5-yr. exposure. These results clearly demonstrate the beneficial effect of the calcined shales.

The remaining slabs made with cements of lower alkali content, 0.70 per cent or less as Na_2O, show no evidence of pattern cracking after 5-yr. exposure. This is true even when the cements were used with the reactive aggregate and without the calcined shale admixtures.

Concrete Beams:

In a later series of tests a number of concrete beams were prepared for outdoor storage. In this study two 6 by 6 by 30-in. beams for each condition of test were prepared and cured moist for 7 days in the laboratory. They were then taken to the Naperville farm where one beam was laid on the ground and the other was set upright in the soil so that about one-half its length was exposed to the weather.

In the preparation of the beams Elgin, Ill., sand and gravel were used as the neutral aggregate, and 95 per cent Elgin sand plus 5 per cent siliceous magnesian limestone with Elgin gravel was the reactive aggregate. Five different cements were used having total alkali contents ranging from a high of 1.05 to a low of 0.21 per cent calculated as Na_2O.

Six different admixtures were used in amounts equal to 15 per cent by weight of the cement. The cement content was maintained constant by using the same quantity of admixture in all mixes. Powdered quartz was used as the control admixture because previous studies had shown that it was not effective in preventing the expansion of specimens made with reactive sands and cements of relatively high alkali content. The mix was 1:2:3.2 by weight calculating the admixture as part of the sand.

The essential characteristics of the composition of the beams are shown in the accompanying Table VIII. Observations of the appearance of pattern cracking after 4 yr. of outdoor storage are also shown in the table.

None of the beams made with the nonreactive aggregate (Elgin sand and gravel alone) shows any evidence of pattern cracking. The beams made with the two cements of highest alkali content, and the reactive aggregate, containing 5 per cent siliceous magnesian limestone, show pronounced pattern cracking. Other beams made with the reactive aggregate and cements of lower alkali content show no evidence of pattern cracking.

The different powdered admixtures gave varying results when used with these cements and the reactive aggregate as follows:

Calcined Monterey shale prevented the development of pattern cracking with the two cements of highest alkali content, 1.05 and 0.92 per cent total alkalies as Na_2O, Nos. 33 and 26.

The calcined clay-lime mixture prevented the pattern cracking with the cement having 0.92 per cent total alkalies, No. 30. It was not used with the cement of highest alkali content.

Fresno pumicite did not prevent the pattern cracking with the cement of highest alkali content, 1.05 per cent, No. 34, though it was effective in preventing the cracking with the cement having 0.92 per cent alkalies, No. 27.

Calcined Mowry shale did not prevent the development of map cracking with the cement having 0.92 per cent alkalies, No. 29. It was not used with the cement of highest alkali content.

Kansas pumicite did not prevent the pattern cracking with the cement having 0.92 per cent alkalies, No. 28, and it actually aggravated the cracking with cements having 0.68 and 0.53 per cent alkalies respectively, Nos. 38 and 41.

These results confirm the findings reported by Mr. Hanna. Some of the pozzolanic materials have shown encouraging results. In tests up to 5 yr. they have prevented the abnormal expansion and pattern cracking usually associated with the alkali-aggregate reaction when they were used with cements of high alkali content and in amounts that might be feasible in the manufacture of portland-pozzolana cements. On the other hand, some of the admixtures, also considered to be pozzolanic materials, gave very discouraging results. They did not appreciably reduce the expansion and pattern cracking associated with the alkali-aggregate reaction and in some cases they actually aggravated the pattern cracking.

These results indicate the need for caution in the use of pozzolanas in concrete. Much further work will be required before it will be possible to write adequate specifications for these materials.

MR. W. C. HANNA (*author*).—Mr. Lerch has shown a table in which he has noted that some of the pozzolanas did not work satisfactorily. My recommendation would be to vary the amount of pozzolana, as we know of no certain percentage to give best results. Other changes might be made to correct this trouble such as changes in temperature, fineness, and water.

MR. LERCH.—Mr. Hanna suggested that when a pozzolan does not give beneficial results it should be used in larger amounts. In many cases that is true, there is some minimum amount of pozzolan required to prevent the abnormal expansion and pattern cracking. However, there are some exceptions. For example, in Table VIII of the discussion it was shown that with quartz silica there was no evidence of pattern cracking with the reactive aggregate for cements Nos. 37 and 40; when Kansas pumicite was substituted for the silica pronounced pattern cracking occurred, cements Nos. 38 and 41. Here is a case where the powdered admixture caused the pattern cracking. It is not likely that larger additions of this admixture would prove beneficial.

MR. DAVIS.—Time permitting, I should like to emphasize by example my earlier statement that all finely divided siliceous materials are reactive with lime. In our investigations of blended cements, previously mentioned, one method of measuring pozzolanic activity consisted of determining the compressive strength at 7 and 28 days of 2 by 4-in. mortar cylinders, composed of two parts of high-calcium hydrated lime, to one part of finely divided siliceous material, to nine parts of Ottawa sand, measured by weight. The specimens were cast in tin cans which were then sealed and cured at the elevated temperature of 130 F. until a short time before testing, when the cans were stripped from the specimens. The mortar cylinders which exhibited the lowest compressive strength at 28 days (139 psi.) were those for which the siliceous material was ground Ottawa sand all passing the No. 325 sieve. After 28 days, companion specimens in their sealed tins were removed from the 130 F. curing cabinet and were placed in the air of the laboratory. When tested at the age of 6 yr., these specimens exhibited a compressive strength of 2580 psi.

MR. WALTER H. PRICE.[6]—Both Mr. Hanna and Mr. Conrow mentioned that they obtained expansion in laboratory specimens made with cements having very low alkali contents. I should like to know whether anyone knows of any concrete structure that is showing expansion which can be attributed to alkali aggregate reaction in which the alkali content of the cement used was below 0.3 per cent?

MR. HANNA.—I do not know that I have ever seen a concrete structure made with cement as low as 0.3 per cent, so certainly I cannot point to a bad one. A cement with 0.3 per cent of alkali would be uncommon. However, good structures made with cements of high-alkali content have been observed.

MR. CONROW.—I cannot say that I know of any structures in which a low-alkali cement was used that have given the trouble.

However, it appears that a potential condition can arise that could give trouble though low-alkali cement were used. The point I wished to make in my discussion was that possibly these alkalies are activators or accelerators and they are causing this other reaction to act faster.

MR. H. F. GONNERMAN.[7]—Would Mr. Conrow please amplify his written discussion with respect to the treatment of the specimens that showed the high expansions. Were they put through the cycle of treatment where you raised the temperature? If so, I think that cycle should be explained.

MR. CONROW.—In my discussion I mentioned the discovery of a way to develop the disease. It was discovered, somewhat by accident, that if the concrete is exposed to relatively high temperatures, not greater, however, than could occur in nature, for a considerable period, say, ten days, that it apparently brought about some condition within that concrete that causes this reaction to become accelerated and the concrete to grow quite rapidly. It is not necessary for us to wait for four or five years to get these results.

The time required for conclusive results was approximately 165 days; some of the expansions had reached values close to 0.21 per cent.

MR. GONNERMAN.—I believe Mr. Conrow stated that when he tested concrete containing limestone aggregate he did not obtain the high expansions, but obtained them only with concrete containing highly siliceous aggregates? Is that correct?

MR. CONROW.—I have tried many aggregates. The limestone aggregate I found to be nonreactive. Any concrete which contained a considerable proportion of the limestone aggregate did not show expansion to a very great degree. It showed expansion, however, because the fine aggregate was a siliceous reactive sand. Apparently, the amount of expansion was not sufficient to cause trouble over the period which the concrete was expected to give service.

I believe the presence of the limestone aggregate probably inhibited the action sufficiently such that concrete made with it gives good service.

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[6] Materials Engineer, U. S. Bureau of Reclamation, Denver, Colo.

[7] Manager, Research Labs., Portland Cement Assn., Chicago, Ill.

POLAROGRAPHIC DETERMINATION OF TETRAETHYL LEAD IN GASOLINE*

By Richard Borup[1,2] and Harry Levin[1]

Synopsis

The tetraethyl lead in a small amount of gasoline is decomposed in a volumetric flask with hydrochloric acid, conducting the operation in such a manner that no quantitative transfer of sample extracts is necessary. The only transfer of sample extract from the original reaction flask is that involved in the actual preparation of the polarogram, and for this purpose a precise measurement of the solution is not necessary. This polarographic determination of tetraethyl lead in gasoline involves a minimum of manipulation, no complex chemical operations, filtrations, washing of precipitate, etc. Because of the few operations, results are easily repeatable. The method is commendable also for its speed—a single sample can be analyzed within 1 hr. and 35 determinations have been made per man-day. Convenient means for controlling temperature and a convenient cell are described.

The subject of tetraethyl lead determination in gasoline is of such importance in the petroleum industry it is little wonder that investigators in this field continue to seek simpler, more reliable, or faster methods for making this determination. Recently Lykken, *et al.* (**1**)[3] described a new chemical approach to the problem and provided an excellent summary of the chemical methods that have been used by others, including the popular A.S.T.M. Standard Method of Test for Tetraethyl Lead in Gasoline (D 526–42),[4] and Gordon and Burdett (**2**) described a microchemical method involving precipitation of lead by iodine ending with thiosulfate titration of chromate equivalent to the lead. Sullivan and Friedman (**3**) described an improved X-ray absorption method for tetraethyl lead in gasoline, employing a Geiger counter to measure radiation, and claimed to complete a determination in 5 min. with an accuracy equivalent to that obtainable by good chemical methods. Frediani and Bass (**4**) briefly described an application of polarographic analysis to the determination of tetraethyl lead in gasoline. The method described here is a refinement and simplification of this polarographic method which, though not as rapid as the X-ray procedure, is faster than strictly chemical methods.

Method of Analysis

Apparatus:

The special apparatus consists of a polarograph, an electrode system, and a constant temperature chamber (Fig. 1).

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[2] Present adress, Foster D. Snell, Inc., New York, N. Y.

[3] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1016.

[4] 1946 Book of A.S.T.M. Standards, Part III-A, p. 287.

Reagents:

Concentrated hydrochloric acid.
Gelatin, 0.05 per cent in water.

Procedure:

Introduce 10 ml. of concentrated hydrochloric acid into a 50-ml. pyrex glass-stoppered volumetric flask and add 10.00 ml. of gasoline sample measured from a fine burette; the temperature of the gasoline must be known. Stopper the flask tightly and shake vigorously for 5 min., avoiding splashing the stopper. Allow to stand a minute to permit the liquid to drain from the neck of the flask. Remove the stopper, add a small glass bead, and place the flask on a steam plate for 30 min. of further digestion. Remove from the steam plate and add distilled water until the gasoline-aqueous interface is in the neck of the flask but below the volume marker. With a pipette withdraw and discard the nonaqueous layer. Add 10 drops of 0.05 per cent gelatin solution. Make up to volume (at 25 C.) with distilled water. Transfer some of the extract to the cell and bubble water-saturated nitrogen through the sample to eliminate dissolved oxygen which has an interfering polarogram of its own; a 5-min. bubbling period is generally adequate for this purpose. With a large number of samples on hand, this step can be expedited by use of a multiple cell arrangement, permitting bubbling of other samples with nitrogen

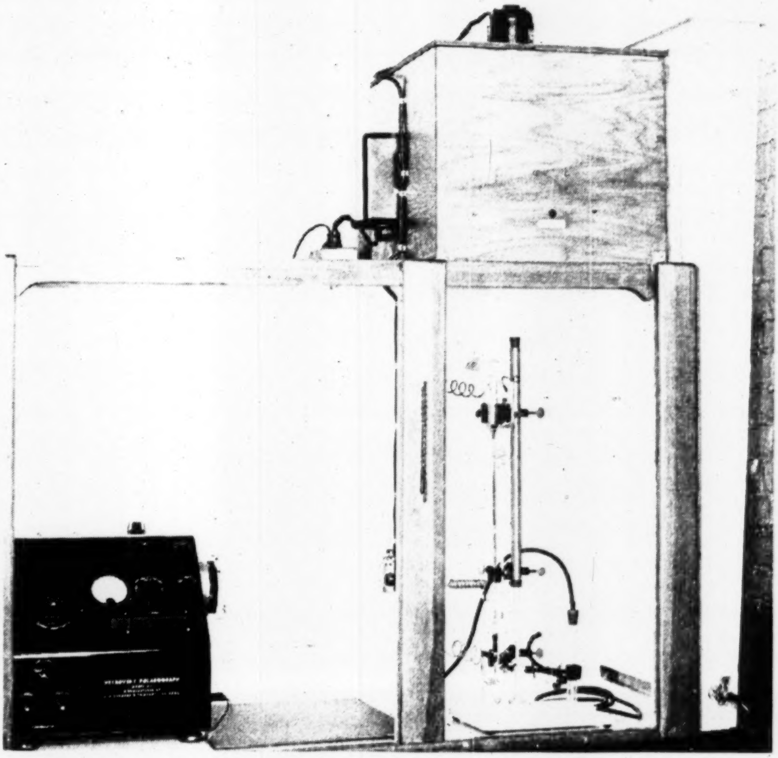

FIG. 1.—Assembly of the Polarograph, the Dropping Mercury Electrode, and the Cell Installed in Their Cabinet.

while the polarogram of one is being formed. Obtain the polarogram between −0.2 and −0.7 v. while sweeping the sample surface only with water-saturated nitrogen.

Derive the tetraethyl lead content by comparing the step height with a working curve prepared from polarograms obtained in a similar manner on samples of known tetraethyl lead content.

TABLE I.—EFFECT OF SHAKING AND HEATING.

Sample	Tetraethyl Lead, ml. per gal.			
	Polarographic Visual[a]		Chemical Method	Prepared
	A	B		
No. 1	2.03	0.15	2.06	....
No. 2	0.81	0.06	0.81	....
No. 3	4.00	0.83	4.01	4.0

[a] "A" runs were shaken for 5 min. and heated for 30 min. "B" runs were only shaken for 5 min.

TABLE II.—EFFECT OF PERIOD OF HEATING.[a]

Sample unknown but containing approximately 1.6 ml. of tetraethyl lead per gallon of gasoline.

Time on Steamplate	Polarographic Values	
hr., min.	Recorded Step at 25.0 C., mm.	Tetraethyl lead, ml. per gal.
– 10	640	1.58
– 20	650	1.62
– 30	660	1.64
– 40	660	1.64
2 15 (to dryness)	589	1.46
2 45 (to dryness)	412	1.02

[a] See also values in Table III obtained when using a different capillary.

The instrument used in our work was a Heyrovsky Polarograph, model XII, manufactured by E. H. Sargent and Co. of Chicago. The ground-glass screen, graduated in millimeters and located on the face of the instrument, permits an operator to observe the galvanometer deflections simultaneously with their being recorded on photographic paper. Thus the operator has an extremely rapid means for making quantitative measurements visually, when speed is essential, while making a permanent photographic record to be developed later. The screen observation is satisfactory for all ordinary purposes.

Working curves were made by running samples of known tetraethyl lead content and plotting the step heights of their polarograms at 25 C. against tetraethyl lead content. Two curves were made, one derived from millimeters of step *observed* on the visible ground-glass scale, and the other from the millimeters of step *measured* on the photographic record.

TABLE III.—EFFECT OF PERIOD OF HEATING.[a]

Sample prepared to contain 4.00 ml. of tetraethyl lead per gallon of gasoline.

Run	Time on Steamplate	Polarographic Step at 25.0 C., mm.		Tetraethyl lead, ml. per gal.	
	hr., min.	Visual	Recorded	Visual	Recorded
No. 1	– 0	420	360	0.83	0.78
No. 2	– 5	1240	1200	2.54	2.56
No 3	– 10	1700	1666	3.50	3.57
No. 4	– 15	1880	1840	3.88	3.97
No. 5	– 20	1920	1860	3.98	4.00
No. 6	– 25	1920	1880	3.98	4.02
No. 7	– 30	1920	1860	3.98	4.00
No. 8	– 40	1920	1860	3.98	4.00
No. 9	– 50	1940	1880	4.00	4.02
No. 10	1 –	1920	1860	3.98	4.00
No. 11	1 10	1920	1860	3.98	4.00
No. 12	1 20	1920	1860	3.98	4.00

[a] See also values in Table II obtained when using a different capillary.

DISCUSSION AND EFFECT OF VARIABLES

In testing the polarographic method, the following variations from the aforedescribed gasoline decomposition procedure were tried: (*a*) not heating at all after the shaking period, (*b*) heating for different lengths of time, and (*c*) evaporating to dryness. It was found (Table I) that shaking alone was not sufficient and that a heating period was necessary, although less than 30 min. may be sufficient (Tables II and III). Evaporating to dryness required more than 2 hr. and gave low results compared to the 30-min. heating period (Table II).

A test was made to determine whether heating the volumetric flasks on the steam plate introduced any error due to permanent changes in calibration effected thereby. The volume changes were of the order of 0.01 per cent; hence negligible.

Tests were also made to determine whether the presence of alcohol, benzene, xylidine, formaldehyde, unsaturated hydrocarbons, and gasoline gum affected the quantitative recovery of lead from the tetraethyl lead in the gasoline. It was found (Tables IV and V) that their presence did not interfere. Incidentally, the presence of some of these substances greatly complicates chemical methods of determination. Increasing the volatility of a leaded gasoline by adding 50 per cent of isopentane had no effect on the polarographic result computed on the diluted basis.

TABLE IV.—DATA ON COMPLEX SAMPLES.

Sample[a]	Tetraethyl Lead, ml. per gal.	
	Polarographic Visual	Chemical Method
A	1.85 1.88	1.90 1.90
B	1.88 1.88	1.90 1.90
C	None None	None None

[a] A = 28 per cent by volume benzene
2 per cent by volume xylidene
1 per cent by volume formaldehyde
19 per cent by volume alcohol
50 per cent by volume gasoline
} Prepared to contain 1.9 ml. of tetraethyl lead per gallon

= 28 per cent by volume benzene
2 per cent by volume xylidene
20 per cent by volume alcohol
50 per cent by volume gasoline
} Prepared to contain 1.9 ml. of tetraethyl lead per gallon

C = 1 N HCl plus 2 per cent xylidene in gasoline (no tetraethyl lead)

Temperature plays an important part in the determination. A correction must of course be made for the temperature of the gasoline at the time of sampling. Provision for this is made in the A.S.T.M. Method D 526[4] which specifies that the results in milliliters of tetraethyl lead per gallon at the observed temperature must be corrected to 60 F. (15.5 C.) by applying a correction to the milliliters of tetraethyl lead per gallon found, as follows:

$$\text{Percentage correction} = 0.1\ (t - 15.5\ \text{C.})$$

where t is the observed temperature of the sample in degrees Centigrade. In addition, during the making of the polarogram, the temperature must be maintained within ± 0.5 C. to control the effects due to temperature within ± 1.0 per cent (6). For this purpose the entire electrode-cell portion of the apparatus is enclosed in a cabinet (Fig. 1) whose temperature is maintained at 25 ± 0.5 C. During the short time required to make the polarogram the temperature varies less than 0.2 C.

TABLE V.—GASOLINE OF HIGH GUM OR UNSATURATION.

Sample	Gum, mg. per 100 ml.		Bromine Number	Tetraethyl Lead, ml. per gal.	
	A.S.T.M.[a]	Copper Dish[b]		Polarographic Recorded	Chemical Method
No. 1	10	67	62	$\left.\begin{matrix}3.47\\3.47\end{matrix}\right\}3.47$	$\left.\begin{matrix}3.54\\3.58\end{matrix}\right\}3.56$
No. 2	266	309	46	$\left.\begin{matrix}2.53\\2.49\end{matrix}\right\}2.51$	$\left.\begin{matrix}2.42\\2.45\end{matrix}\right\}2.44$
No. 3	1	16	52	$\left.\begin{matrix}2.95\\2.99\end{matrix}\right\}2.97$	2.95
No. 4	2	19	53	$\left.\begin{matrix}3.01\\2.99\end{matrix}\right\}3.00$	3.00

[a] Standard Method of Test for Existent Gum in Gasoline (Air-Jet Evaporation Method) (D 381 – 46), 1946 Book of A.S.T.M. Standards, Part III-A, p. 210.

[b] Gum Content of Gasoline (Copper Dish Method) (United States Government Method 330.11), Federal Standard Stock Catalog, Section IV, Part 5, VV-L-791c, May 12, 1945, p. 149.

The glass beads, added to prevent bumping while heating, have an average volume of 0.020 to 0.025 ml. Thus one bead in a 50-ml. volumetric flask, if ignored, introduces an error of 0.05 per cent which may be considered insignificant.

Capillaries broke in the course of the

work and new ones were prepared. The polarograms of unknowns should of course be compared with calibration curves derived from polarograms of knowns run in the same capillary as the unknown since different capillaries may yield different step height coefficients. Table VI is a sample of calibration data for a specific capillary.

With a large number of samples on hand as many as 35 tetraethyl lead determinations have been made per man-day. The elapsed time for a single test is about 1 hr. and a sample in duplicate requires about 10 min. longer. These estimates of time are for visual (observed) measurements. Recorded (photographic) results add about an hour longer for a single sample and little more for a group of eight determinations which can all be done on one piece of photographic paper.

TABLE VI.—TYPICAL CALIBRATION DATA.

Polarographic Step at 25.0 C., mm.		Tetraethyl Lead, ml. per gal.	
Visual	Recorded	Prepared	Chemical Method
2900	2800	6.0	6.04
2420	2340	5.0	5.07
1940	1830	4.0	4.08
1460	1410	3.0	3.09
980	940	2.0	2.08
500	470	1.0	1.09
260	245	0.5	0.56
80	66	0.1	0.13
29	None	None	None

Statistical analysis of the data obtained in the examination of 50 samples taken at random and run in duplicate, of 10 determinations made on one sample, and of 31 samples (Table VII) run both polarographically and chemically, indicate that the precision of the polarographic results obtained from the recorded polarograms is not significantly different from the precision of the results obtained by a chemical method. The chemical method used for comparison involved decomposition by A.S.T.M. Method D 526,[4] and ended in thiosulfate (iodimetric) titration of chromate equivalent to the lead. The precision of this chemical method fulfills the requirement of A.S.T.M. Method D 526. Statistical analysis of the aforementioned data also indicates that the polarographic results obtained from the visually observed galvanometer deflections, if multiplied by 0.99 yield corrected values having the same correlation with chemical results as do those obtained from the recorded polarograms. The upper control limit for the polarographic results is 0.062 ml. of tetraethyl lead per gallon, corresponding to an average spread of 0.019 in the results. That is, if the spread between duplicate tests made polarographically is greater than 0.062, the determinations should be repeated.

TABLE VII.—COMPARISON OF RESULTS ON UNKNOWNS BY POLAROGRAPHIC AND CHEMICAL METHODS (ROUTINE APPLICATIONS).

Tetraethyl Lead, ml. per gal.

Polarographic		Chemical (*c*)	Difference (*c*) − (*b*)
Visual (*a*)	Recorded (*b*)		
None	None	None	
None	None	None	
None	None	None	
None	None	None	
None	None	None	
0.21	0.25	0.19	−0.06
0.28	0.28	0.21	−0.07
0.53	0.56	0.59	0.03
0.63	0.59	0.57	−0.02
0.76	0.78	0.84	0.06
0.84	0.81	0.80	−0.01
0.86	0.81	0.82	0.01
1.05	1.02	1.05	0.03
1.06	1.06	1.06	0
1.16	1.11	1.14	0.03
1.20	1.20	1.25	0.05
1.23	1.22	1.27	0.05
1.27	1.27	1.29	0.02
1.29	1.31	1.26	−0.05
1.58	1.52	1.56	0.04
1.60	1.58	1.57	−0.01
1.62	1.57	1.62	0.05
1.70	1.66	1.65	−0.01
1.94	1.94	1.91	−0.03
2.00	2.02	1.98	−0.04
2.02	2.00	2.00	0
2.06	2.10	1.98	−0.12
2.28	2.28	2.37	0.09
2.66	2.57	2.62	0.05
3.08	2.98	3.00	0.02
3.10	3.10	3.04	−0.06

The polarographic data used in the above statistical analysis was accumulated when the instrument was in a location subject to frequently disturbing vibrations. Very recently a much more favorable location for the polarograph has become available and the lesser amount of data already accumulated there show an upper control limit of 0.045, corresponding to an average spread of 0.014 in the results, hence one may expect a precision satisfying the A.S.T.M. requirement.

TABLE VIII.—POLAROGRAPHIC RESULTS ON REDUCED VOLUME SAMPLES.

Sample[a]	Tetraethyl Lead, ml. per gal.		
	10 ml. Sample	0.5 ml. Sample	0.1 ml Sample
No. 1	3.69	3.70	....
No. 2	2.83	2.58	
No. 3	3.09	....	3.37

[a] These samples were three different products.

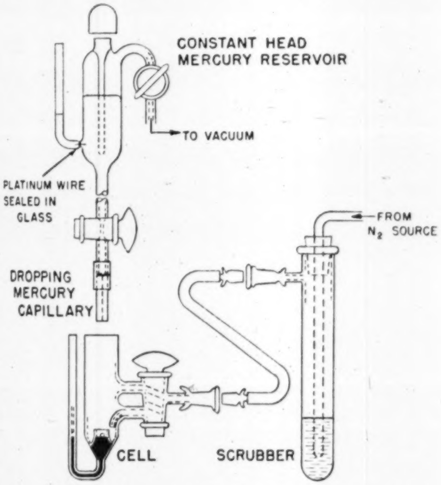
CONSTANT HEAD
MERCURY RESERVOIR
TO VACUUM
PLATINUM WIRE
SEALED IN
GLASS
FROM
N_2 SOURCE
DROPPING
MERCURY
CAPILLARY
CELL
SCRUBBER

FIG. 2.—Construction of the Dropping Mercury Electrode and Cell.

Though 10 ml. of gasoline sample has been employed generally in the analyses reported, tests made on smaller samples demonstrate its utility on what relatively may be considered micro quantities. The results in Table VIII are illustrative. Further refinements in technique of handling the small samples will no doubt improve the precision.

Figure 1 shows the assembly of the polarograph, the dropping mercury electrode, and the cell installed in their cabinet. The sliding doors with which the cabinet is equipped have been removed for purpose of this photograph. The temperature of the right-hand com-

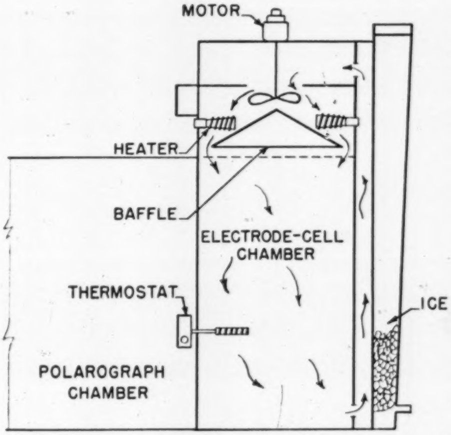
MOTOR
HEATER
BAFFLE
ELECTRODE-CELL
CHAMBER
THERMOSTAT
ICE
POLAROGRAPH
CHAMBER

FIG. 3.—Constant Temperature-Control Unit.

FIG. 4.—A Typical Polarogram.

partment of the cabinet is thermostatically controlled by delivery of cooled or heated air as shown.

The construction of the mercury electrode and cell is illustrated in Fig. 2. Note the Mueller (5) cell for providing a constant head of mercury.

Figure 3 shows the manner in which the temperature is controlled where needed in the cabinet.

A typical polarogram is shown in Fig. 4.

Acknowledgment:

The authors express their appreciation to J. Furtnett whose careful work provided much of the data, to J. Sherman for his statistical analyses and criticism, and to C. J. Anderson for his valuable assistance in preparing and reviewing the manuscript.

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HARDNESS AND ABRASION RESISTANCE OF PLASTICS*

BY L. BOOR,[1] J. D. RYAN,[2] M. E. MARKS,[3] AND W. F. BARTOE[4]

SYNOPSIS

Representative samples of typical commercial plastics were tested by several procedures, for properties associated with the descriptive term "hardness." Included were various indentation, scratch, and wear methods. At least five aspects of behavior, each one contributing in various degrees to the test value obtained by any given method, are indicated.

DURING the past two years, Subcommittee II on Hardness of the A.S.T.M. Committee D-20 on Plastics has been investigating various aspects of the property termed "hardness" as applied to plastics. A group of typical materials was collected, divided among several cooperating laboratories, and evaluated by the various procedures described. The test data were tabulated and an attempt was made to relate the various indices of "hardness."

The materials tested are listed in Table I.

The various contributing laboratories are identified as follows: L.O.F., Libbey-Owens-Ford Glass Co.; P.P.G., Columbia Chemical Division, Pittsburgh Plate Glass Co.; ACCO, American Cyanamid Co.; and R & H, Rohm & Haas Co.

TEST METHODS

The various test procedures used were as follows:

Knoop Hardness Number:

The instrument used was the Tukon Tester, made by Wilson Mechanical Instrument Co., New York, N. Y. The Knoop Indenter has been described elsewhere.[5, 6] The Knoop hardness number is the ratio of load in kilograms to the unrecovered projected area of indentation in square millimeters. The long dimension of the indentation was measured with a microscope and the corresponding indentation number was determined from a table supplied with the instrument.

Rockwell Hardness Numbers:

The standard Rockwell Hardness Tester, made by Wilson Mechanical Instrument Co., was used. The

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[3] Columbia Chemical Division, Pittsburgh Plate Glass Co., Barberton, Ohio.

[4] Rohm & Haas Co., Philadelphia, Pa.

[5] Frederick Knoop, Chauncey G. Peters, and Walter B. Emerson, "A Sensitive Pyramidal-Diamond Tool for Indentation Measurements," Nat. Bureau Standards, *Research Paper RP 1220*, July, 1939.

[6] Vincent E. Lysaght, "The Knoop Indenter as Applied to Testing Nonmetallic Materials, Ranging from Plastics to Diamonds." ASTM BULLETIN No. 138, January, 1946, p. 395.

TABLE I.—MATERIALS TESTED.

	Material	Type	Source
No. 1	Plexiglas IA—⅛ in.	Methyl methacrylate	Rohm & Haas Co.
No. 2	Plexiglas IA—¼ in.	Methyl methacrylate	Rohm & Haas Co.
No. 3	Plexiglas II—⅛ in.	Methyl methacrylate	Rohm & Haas Co.
No. 4	Plexiglas II—¼ in.	Methyl methacrylate	Rohm & Haas Co.
No. 5	Lucite HC201 (Std.) R3393—⅛ in.	Methyl methacrylate	E. I. duPont de Nemours & Co., Inc.
No. 6	Lucite HC202 ("Boilable") R-3394—⅛ in.	Methyl methacrylate	E. I. duPont de Nemours & Co., Inc.
No. 7	CR-39-1 ZS 3364	Allyl polymer	Columbia Chemical Div. Pittsburgh Plate Glass Co.
No. 8	Laminac 3414 (clear)	Unsaturated polyester resin	American Cyanamid Co.
No. 9	Laminac 3323 (canvas laminate)	Unsaturated polyester resin	American Cyanamid Co.
No. 10	Vinylite VS 1310 (clear)	Vinyl resin	Bakelite Corp.
No. 11	Polystyrene XMS 10023 (clear compression molded)	Polystyrene	Bakelite Corp.
No. 12	Polystyrene F3 (clear injection molded)	Polystyrene	Monsanto Chemical Co.
No. 13	Melamine (Kraft paper laminate)	Melamine resin	American Cyanamid Co.
No. 14	Bakelite BM 120	Phenolic resin, wood flour filler	Bakelite Corp.
No. 15	Bakelite BM 261	Phenolic resin, mineral filler	Bakelite Corp.
No. 16	Nylon FM 1(L0139)	Nylon	E. I. duPont de Nemours & Co., Inc.
No. 17	Nitron No. 2272 D1223	Cellulose nitrate	Monsanto Chemical Co.
No. 18	Fibestos No. 2050 TVA D1224	Cellulose acetate	Monsanto Chemical Co.
No. 19	Tenite II 205A 12605 H2	Cellulose acetate-butyrate	Tennessee Eastman Corp.
No. 20	Tenite II 205A 12605 M	Cellulose acetate-butyrate	Tennessee Eastman Corp.
No. 21	Tenite II 205A 12605 S2	Cellulose acetate-butyrate	Tennessee Eastman Corp.
No. 22	Ethyl cellulose 22630 hard	Ethyl cellulose	Celanese Corp.
No. 23	Ethyl cellulose 22631 medium hard	Ethyl cellulose	Celanese Corp.
No. 24	Saran (compression molded)	Vinylidene chloride	Dow Chemical Co.
No. 25	Saran B115 (injection molded)	Vinylidene chloride	Dow Chemical Co.
No. 26	Plate glass		Libbey-Owens-Ford Glass Co.

various scales and methods of test were as follows:

Alpha Scale.—This method of using the Rockwell Instrument has been described by Boor.[7] The ½-in. indenter was used with 60-kg. load applied for 15 sec. The Alpha hardness number is 150 minus the scale divisions of indentations under major load, corrected for the spring of the frame.

Beta Scale.—Same as above except that the major load was 30 kg.

M Scale (ACCO) Method A.S.T.M. D 229-43.[8]—Indenter (¼ in.). 100-kg. major load. Major load was removed 7 to 8 sec. after tripping of handle; hardness reading was made 45 sec. thereafter.

M Scale (P.P.G.-1) Method Federal Specification L-P-406.—Indenter (¼ in.). 100-kg. major load. Zero setting was made 30 sec. after application of minor load. Major load was applied and removed 1 sec. after crank stopped. Hardness reading was made 30 sec. thereafter.

M Scale (P.P.G.-2) Method Federal Specification L-P-406a.—Indenter (¼ in.). 100-kg. major load. Zero setting was made 10 sec. after application of minor load. Major load was applied and removed 7 to 8 sec. after crank was tripped. Hardness reading was made 45 sec. thereafter.

M Scale (R & H) Rohm & Haas Method.—Indenter (¼ in.). 100-kg. major load. Zero setting was made 10 sec. after application of minor load. Major load was applied and removed after 30 sec. Hardness reading was made 30 sec. thereafter.

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[7] L. Boor, "Indentation Hardness of Plastics," *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 969 (1944).

[8] Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229–43), 1944 Book of A.S.T.M. Standards, Part III, p. 405. These methods have since been revised and now appear as D 229–46 in the 1946 Book of A.S.T.M. Standards, Part III-B, p. 36.

TABLE II.—HARDNESS OF PLASTICS (INDENTATION METHODS).

Test Method.........		Tukon Knoop		Rockwell Hardness								Barcol		
		Scale—Knoop No.		Alpha Scale		Beta Scale	M Scale	M Scale	M Scale	M Scale	R Scale	[a]	[a]	[b]
	Material	Laboratory.... LOF	PPG	ACCO	PPG	ACCO	ACCO	PPG-1	PPG-2	R & H	ACCO	PPG	R & H	LOF
No. 1..	Plexiglas IA—1/8 in.	...	15.0	102	96	128	97	93	93	72	125	33	35	29
No. 2..	Plexiglas IA—1/4 in.	16.0	14.9	100	96	127	102	98	94	79	125	37	35	..
No. 3..	Plexiglas II—1/8 in.	...	14.7	107	104	129	107	102	105	88	126	45	40	34
No. 4..	Plexiglas II—1/4 in.	17.1	16.0	102	98	126	107	103	103	89	126	44	42	..
No. 5..	Lucite HC201—1/8 in.	16.0	15.6	104	100	129	98	97	99	78	125	36	35	26
No. 6..	Lucite HC202—1/8 in.	18.1	18.1	108	102	129	107	104	106	90	126	47	45	36
No. 7..	CR-39-1	16.1	12.1	94	92	124	100	99	99	87	123	31	27	28
No. 8..	Laminac clear	13.0	10.6	91	89	122	97	94	91	72	124	25	20	21
No. 9..	Laminac canvas laminate	...	..	75	72	114	...	40	37	..	98	15	..	22
No. 10..	Vinylite clear	13.8	14.1	105	103	130	...	74	77	60	123	19	17	12
No. 11..	Polystyrene compression	16.8	15.6	106	104	130	86	83	80	69	125	30	27	19
No. 12..	Polystyrene injection	17.1	16.7	109	105	132	78	73	87	66	124	30	28	25
No. 13..	Melamine laminate	62.0	..	130	126	140	126	126	126	..	129	69	..	69
No. 14..	Bakelite BM 120	42.4	..	122	121	137	118	116	115	..	127	58	..	55
No. 15..	Bakelite BM 261	52.9	..	128	113	140	111	109	110	..	124	57	..	57
No. 16..	Nylon	15.0	..	102	95	128	87	82	79	..	120	8	..	2
No. 17..	Nitron	13.2	12.6	78	75	119	...	48	42	23	112	4	0	0
No. 18..	Fibestos	12.1	7.8	68	53	113	...	64	56	25	115	0	0	0
No. 19..	Tenite II H2	7.5	7.1	59	56	109	...	50	44	−21	110	0	0	0
No. 20..	Tenite II M	5.9	5.6	37	32	100	...	37	27	−44	99	0	0	0
No. 21..	Tenite II S2	4.7	4.2	6	...	87	...	26	12	−64	81	0	0	0
No. 22..	Ethyl cellulose, hard	8.8	8.2	61	55	110	...	53	48	5	109	0	0	0
No. 23..	Ethyl cellulose, medium	6.0	5.9	43	37	101	...	51	41	12	102	0	0	0
No. 24..	Saran compression	6.4	5.2	38	35	97	...	45	40	..	92	0	0	0
No. 25..	Saran injection	3.8	..	12	...	86	...	46	42	..	86	0	0	0
No. 26..	Plate glass	480	..	...	...	...	...	...	...	..	...	..	..	98+

[a] Maximum readings.

[b] Readings made 10 sec. after application of load.

Only transparent samples were tested.

R Scale Method A.S.T.M. D 229–43.[8]—Indenter ($\frac{1}{2}$ in.). 60-kg. major load. Major load was removed 7 to 8 sec. after tripping of handle; hardness reading was made 45 sec. thereafter.

Barcol Hardness:

Barber Colman Co., Rockford, Ill. Sharp point indenter, spring-loaded. Readings made as indicated in Table II.

Shore Scleroscope Hardness:

Shore Instrument and Mfg. Co., Jamaica, N. Y. Falling tup. Relative height of rebound.

Sward Rocker Hardness:

Used in the paint and varnish industry to rate relative hardness.[9] The rocker is caused to oscillate on the test surface, and the number of rocks between two fixed angles of amplitude is a measure of surface hardness.

Number of rocks times 2 = Sward Hardness. Glass = 100.

Bierbaum Scratch Hardness:

Bierbaum Microcharacter, American Optical Co., Buffalo, N. Y. The corner of an accurately ground diamond cube is drawn over the test surface with a 3-g. load. The width of scratch is measured with a microscope.

$$\text{Bierbaum Scratch Hardness} = \frac{\text{Load (kg.)}}{W^2}$$

where W = width of scratch in millimeters.

Kohinoor Value:

Sharpened pencils were used ranging in hardness from soft to hard as follows:

B–HB–F–H–2H–3H—etc., to 9*H*

The hardness index was the numbered pencil which first showed a visible mark on the test surface.

Moh Value:

Sharp edges of the minerals of the Moh scale were drawn across the surface of the plastic under light pressure. The softest mineral which gave a visible scratch is indicated.

Mar resistance:

A.S.T.M. Method D 673–44.[10]

Relative reduction in gloss produced by various amounts of No. 80 carborundum falling 25 in. through a tube, with the test surface inclined at 45 deg. to the axis of the tube.

Falling Emery Abrasion:

M60 emery (American Optical Co.) was dropped 66 in. through a tube $\frac{7}{8}$ in. in diameter onto the test surface at 45 deg. to the axis of the tube. The standards of comparison were Plexiglas I and Plate Glass. Haze was measured according to A.S.T.M. Method D 672–44T[11] (Kline-Bowen Haze Meter). Since this measures light transmission, only the transparent samples were tested. Haze in the range 5 to 30 per cent was assumed to be in direct proportion to the amount of abrasive. Then per cent haze/grams of

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[9] H. A. Gardner and G. G. Sward, "Physical and Chemical Examination of Paints, Varnishes, Lacquers and Colors," Henry A. Gardner Labs., Bethesda, Md., tenth edition, (1946).

[10] Standard Method of Test for Mar Resistance of Plastics (D 673–44), 1946 Book of A.S.T.M. Standards, Part III-B, p. 209.

[11] Tentative Method of Test for Haze of Transparent Plastics by Photoelectric Cell, 1944 Book of A.S.T.M. Standards, Part III, p. 1653. These Tentative Methods have since been revised and appear as D 672–45 T in the 1946 Book of A.S.T.M. Standards, Part III-B, p. 870.

TABLE III.—EVALUATION OF PLASTICS BY TESTS RELATED TO HARDNESS.

Test Method............		Shore Scleroscope	Sward Rocker	Bierbaum Scratch Hardness (3-g. load)			Kohinoor Value		Moh Value
	Material	Laboratory.... LOF	LOF	PPG	R & H	LOF	LOF	R & H	LOF
No. 1..	Plexiglas IA—⅛ in.	80	52	18.9	15.4	17.5	5-6H	8H	2-3
No. 2..	Plexiglas IA—¼ in.	80	52	18.5	15.5	17.5	5-6H	9H	2-3
No. 3..	Plexiglas II—⅛ in.	81	46	18.6	16.4	19.2	5-6H	9H+	2-3
No. 4..	Plexiglas II—¼ in.	81	46	15.0	16.4	19.2	5-6H	8H	2-3
No. 5..	Lucite HC201—⅛ in.	81	40	19.3	15.2	17.7	5-6H	8H	2-3
No. 6..	Lucite HC202—⅛ in.	83	45	37.0	20.2	16.7	5-6H	8H	2-3
No. 7..	CR 39-1	81	50	40.6	17.8	15.3	4-5H	9H	1-2
No. 8..	Laminac clear	81	56	15.2	10.3	13.5	2-3H	4H	2-3
No. 9..	Laminac canvas laminate	60	15	..	..	12.7	2-3H	...	2-3
No. 10..	Vinylite clear	73	54	10.5	9.3	10.1	2-3H	2H	1-2
No. 11..	Polystyrene compression	77	42	11.1	10.5	8.9	2-3H	2H	1-2
No. 12..	Polystyrene injection	71	76	9.6	11.9	10.3	2-3H	2H	1-2
No. 13..	Melamine laminate	102	74	..	..	32.4	9H	...	2-3
No. 14..	Bakelite BM120	86	31	..	..	19.6	7-8H	...	2-3
No. 15..	Bakelite BM261	80	39	..	..	21.2	4-5H	...	2-3
No. 16..	Nylon	79	48	..	..	11.1	2-3H	...	1-2
No. 17..	Nitron	66	31	41.6	8.2	9.8	2-3H	H	1-2
No. 18..	Fibestos	68	36	11.8	8.1	11.3	4-5H	4H	1-2
No. 19..	Tenite II H2	67	48	16.9	5.9	6.3	HB	B	1-2
No. 20..	Tenite II M	56	48	19.1	3.9	5.4	HB	B	1-2
No. 21..	Tenite II S2	50	42	17.6	3.6	3.8	HB	2B	1-2
No. 22..	Ethyl cellulose, hard	64	34	10.0	4.5	5.6	HB-H	B, F, H	1-2
No. 23..	Ethyl cellulose, medium	57	39	8.6	4.7	5.6	HB-H	B, F	1-2
No. 24..	Saran compression	40	20	..	..	8.7	4-5H	...	1-2
No. 25..	Saran injection	38	18	..	..	9.2	3-4H	...	1-2
No. 26..	Plate glass	109	100	..	..	794	9H+	...	4-5

TABLE IV.—FALLING ABRASIVE TESTS ON PLASTICS.

Test Method		Mar Resistance A.S.T.M. D 673-44				Falling Emery Abrasion						
Laboratory............		ACCO				PPG						
		Percentage of Original Gloss[a]				Percentage of Original Haze	Haze, per cent		Increase in percentage of Haze		Ratio[b]	
		Abrasive					Abrasive		Abrasive		To Standard	To Glass
	Materia	400 g.	800 g.	1200 g.	1600 g.		50 g.	500 g.	50 g.	500 g.		
No. 1..	Plexiglas IA—⅛ in.	60.7	50.6	46.0	45.8	1.2	22.0	..	20.8	..	1.0	0.07
No. 2..	Plexiglas IA—¼ in.	62.5	50.6	46.4	44.0	1.7	22.9	..	21.2	..	1.0	0.07
No. 3..	Plexiglas II—⅛ in.	72.5	55.4	50.8	46.2	1.2	19.0	..	17.8	..	0.9	0.08
No. 4..	Plexiglas II—¼ in.	70.0	53.4	48.7	46.5	0.9	18.1	..	17.2	..	0.9	0.09
No. 5..	Lucite HC201—⅛ in.	46.2	40.7	40.7	39.5	1.4	18.0	..	16.6	..	1.0	0.09
No. 6..	Lucite HC202—⅛ in.	69.6	55.0	51.0	46.3	4.6	21.0	..	16.4	..	1.0	0.09
No. 7..	CR 39-1	95.0	93.0	91.5	88.8	1.7	2.7[c]	11.6	1.0[c]	9.9	16.6	1.5
No. 8..	Laminac clear	92.6	89.0	87.2	84.4	14.4	15.8[c]	28.8	1.4[c]	14.4	11.9	1.1
No. 9..	Laminac canvas laminate	88.2	85.0	74.7	52.3	..	...	..	...	..	...	...
No. 10..	Vinylite clear	59.9	53.2	50.9	47.1	1.6	22.4	..	20.8	..	0.8	0.07
No. 11..	Polystyrene compression	39.7	30.2	30.2	33.3	26.7	63.1	..	36.4	..	0.5	0.04
No. 12..	Polystyrene injection	36.8	29.0	28.6	30.1	6.9	43.9	..	37.0	..	0.4	0.04
No. 13..	Melamine laminate	75.5	54.8	49.2	43.6	..	...	..	...	..	...	...
No. 14..	Bakelite BM120	85.0	73.4	64.4	50.3	..	...	..	...	..	...	...
No. 15..	Bakelite BM261	44.9	40.7	38.2	35.4	..	...	..	...	..	...	...
No. 16..	Nylon	51.3	36.5	30.4	31.6	..	...	..	...	..	...	...
No. 17..	Nitron	68.2	59.5	56.5	55.1	5.5	19.8	..	14.3	..	1.2	0.10
No. 18..	Fibestos	78.2	62.0	51.8	43.8	4.2	22.7	..	18.5	..	0.9	0.08
No. 19..	Tenite II H2	61.6	48.6	44.3	47.1	12.2	26.7	..	14.5	..	1.1	0.10
No. 19..	Tenite II M	63.5	55.1	50.7	48.7	10.8	28.0	..	17.2	..	1.0	0.09
No. 21..	Tenite II S2	51.8	50.8	47.7	45.4	10.5	23.8	..	13.3	..	1.2	0.11
No. 22..	Ethyl cellulose, hard	60.0	48.1	41.5	38.4	18.4	36.1	..	17.7	..	0.9	0.08
No. 23..	Ethyl cellulose, medium	65.9	50.3	43.8	41.0	..	28.4	..	...	..	...	...
No. 24..	Saran compression	40.5	29.2	31.3	32.1	..	...	..	...	..	...	...
No. 25..	Saran injection	65.7	50.7	45.4	42.4	..	...	..	...	..	...	...
No. 26..	Plate glass	99	98	97	96	0.9	2.4[c]	15.4	1.5[c]	14.5	11.0	1.0
	Standard (Plexiglas)	..	..	..	..	0.9	17.5	..	16.6	..	1.0	0.09

[a] Original gloss of each sample rated 100 per cent.

[b] Relative amount of abrasive to produce same increase in haze as on standard plexiglas and plate glass, respectively.

[c] Estimated from 1/10 of increase in haze caused by 500 g. abrasive.

TABLE V.—TABER ABRASION TESTS.[a]

		CS10 Wheel						CS17 Wheel		
								Weight Loss, mg.		
	Material	Original Haze, per cent	Revolutions	Haze, per cent	Increase in haze, per cent	Increase per Revolution, per cent	Ratio to Standard[c]	200 Revolutions	5000 Revolutions (Theoretical)[d]	5000 Revolutions (Actual)
No. 1...	Plexiglas IA—1/8 in.	1.2	5	5.5	4.3	0.86	1.0	4.3	108	120.5
No. 2...	Plexiglas IA—1/4 in.	1.7	5	5.5	3.8	0.76	1.0	2.7	68	114.1
No. 4...	Plexiglas II—1/8 in.	1.2	5	5.7	4.5	0.90	1.0	2.3	58	97.9
No. 4...	Plexiglas II—1/4 in.	0.9	5	5.2	4.3	0.86	1.0	3.0	75	103.7
No. 5...	Lucite HC201—1/8 in.	1.4	4	6.4	5.0	1.25	0.7	9.2	230	114.3
No. 6...	Lucite HC202—1/8 in.	4.6	1	10.4	5.8	5.8	0.15	2.5	63	71.2
No. 7...	CR 39-1	1.7	140	9.4	7.7	0.06	14.3	6.2	155	75.0
No. 8...	Laminac clear	14.4	35	8.3	[b]	...	...	4.2	105	173.1
No. 9...	Laminac canvas laminate	..	...	..	...	...	...	4.4	110	379.7
No. 10...	Vinylite clear	1.6	5	6.4	4.8	0.96	0.9	2.7	68	110.7
No. 11...	Polystyrene compression	26.7	1	38.3	11.6	11.6	0.07	21.6	540	259.1
No. 12...	Polystyrene injection	6.9	1	9.1	2.2	2.2	0.39	..	...	...
No. 13...	Melamine laminate	..	...	..	...	...	...	4.8	120	129.7
No. 14...	Bakelite BM120	..	...	..	...	...	...	8.8	220	106.7
No. 15...	Bakelite BM261	..	...	..	...	...	...	18.9	470	382.8
No. 16...	Nylon	..	...	..	...	...	...	1.8	45	31.4
No. 17...	Nitron	5.5	7	11.0	5.5	0.79	1.1	4.6	115	212.5
No. 18...	Fibestos	4.2	10	8.0	3.8	0.38	2.3	2.0	50	142.7
No. 19...	Tenite II H2	12.2	2	9.2	[b]	...	...	10.5	262	259.1
No. 20...	Tenite II M	10.8	2	19.0	8.2	4.1	0.21	16.6	415	415.2
No. 21...	Tenite II S2	10.5	2	17.1	6.6	3.3	0.26	15.2	380	447.7
No. 22...	Ethyl cellulose, hard	18.4	1	14.5	[b]	...	...	16.9	420	285.1
No. 23...	Ethyl cellulose, medium	..	1	15.9	...	...	...	18.5	460	311.8
No. 24...	Saran compression	..	...	..	...	...	...	5.8	145	375.9
No. 25...	Saran injection	..	...	..	...	...	...	1.3	33	275.9
	Standard (Plexiglas)	0.9	5	5.2	4.3	0.86	1.0	..	...	....

[a] Data from PPG Laboratory.
[b] Haze reading after abrasion lower than original.
[c] Ratio to standard. Relative number of revolutions to produce 5 per cent increase in haze over original.
[d] Assuming weight loss 25 times weight loss at 200 revolutions.

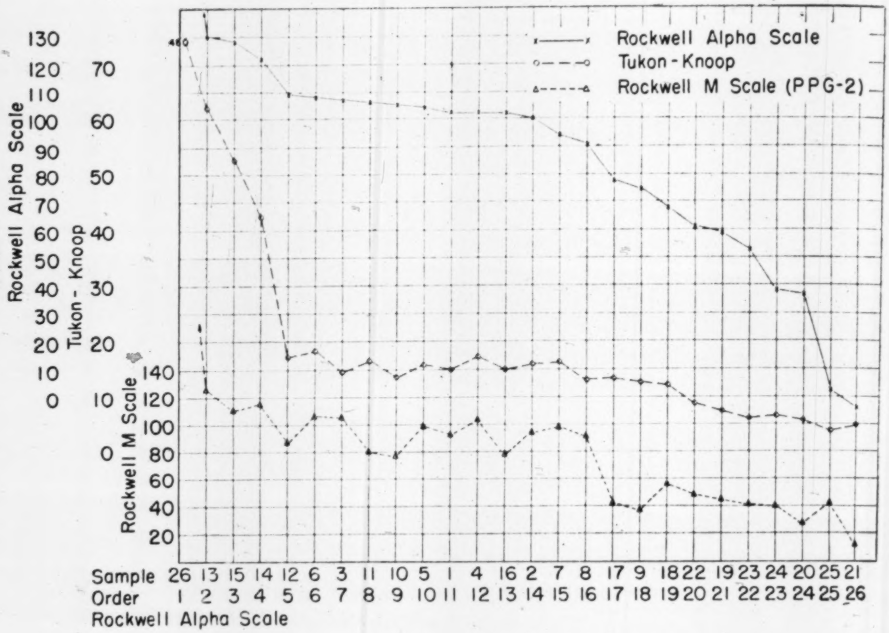
Rockwell Alpha Scale
Tukon - Knoop
Rockwell M Scale
Rockwell Alpha Scale
Tukon - Knoop
Rockwell M Scale (PPG-2)
460
130
120
110
100
90
80
70
60
50
40
30
20
10
0
70
60
50
40
30
20
10
0
140
120
100
80
60
40
20
Sample 26 13 15 14 12 6 3 11 10 5 1 4 16 2 7 8 17 9 18 22 19 23 24 20 25 21
Order 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26
Rockwell Alpha Scale

Fig. 1.

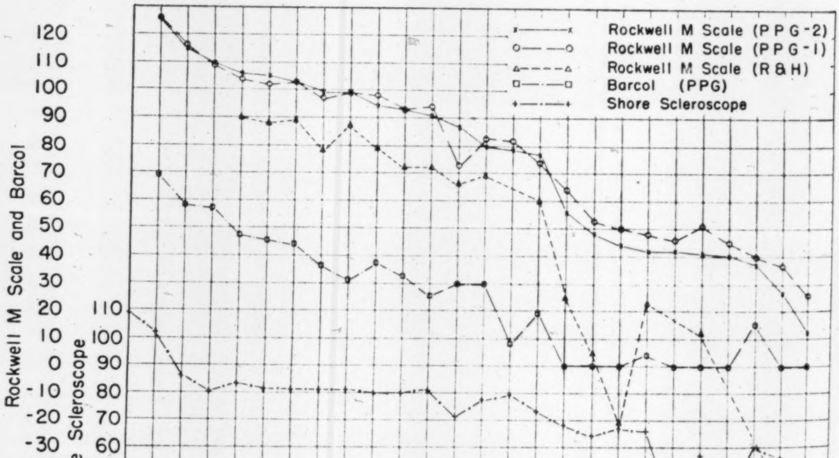
Rockwell M Scale and Barcol
Shore Scleroscope
Rockwell M Scale (P P G -2)
Rockwell M Scale (P P G -1)
Rockwell M Scale (R & H)
Barcol (PPG)
Shore Scleroscope
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
110
100
90
80
70
60
50
40
30
Sample 26 13 14 15 6 3 4 5 7 2 1 8 12 11 16 10 18 22 19 17 25 23 24 9 20 21
Order 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26
Rockwell M Scale

Fig. 2.

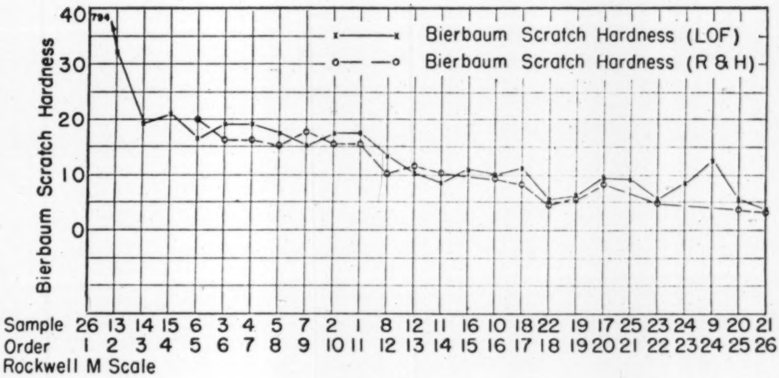

Fig. 3.

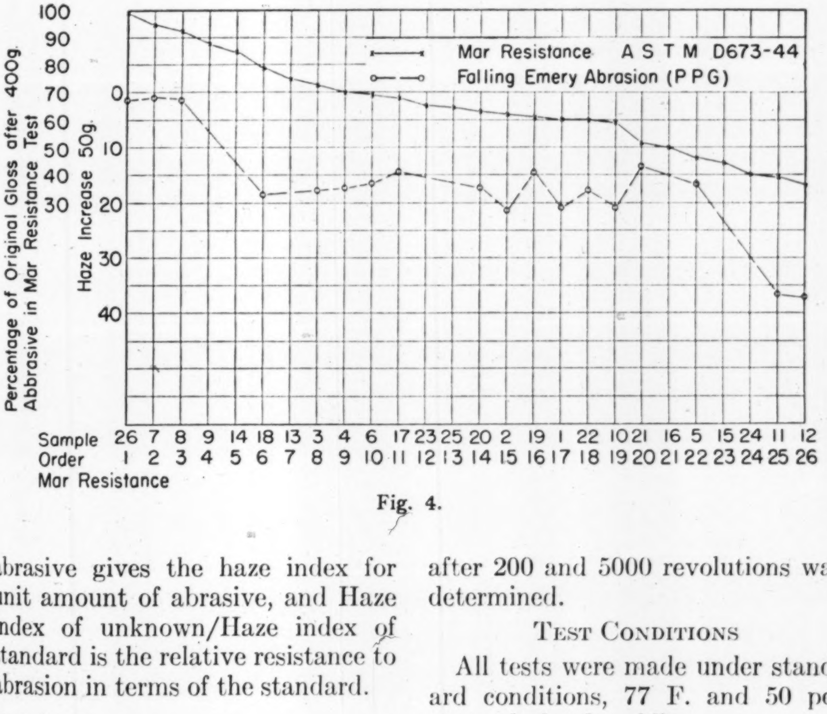

Fig. 4.

abrasive gives the haze index for unit amount of abrasive, and Haze index of unknown/Haze index of standard is the relative resistance to abrasion in terms of the standard.

Taber Abrasion:

Taber Abraser, Taber Instrument Co., North Tonawanda, N. Y.

CS10 and CS17 wheels were used with 1000-g. load. Haze meter was used to measure the abraded track and compared with the standard. The CS17 wheels were used on a fresh set of samples and weight loss after 200 and 5000 revolutions was determined.

Test Conditions

All tests were made under standard conditions, 77 F. and 50 per cent relative humidity.

Test Results

Table II shows the values obtained by various indentation methods.

Table III gives values obtained by scleroscope, Sward rocker, Bierbaum, Kohinoor value, and Moh index.

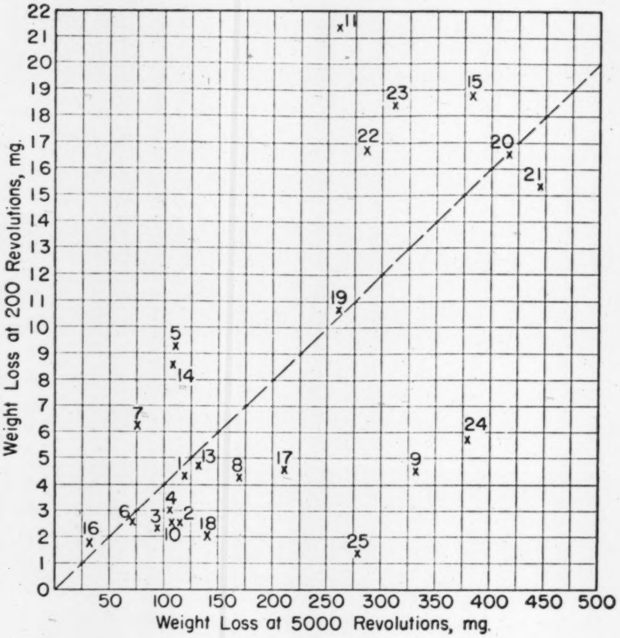

Fig. 5.

Table IV gives values obtained by the two falling abrasive methods.

Table V shows results obtained on the Taber Abraser.

To arrive at the interrelation of the various indices of hardness, many graphs were made of which the more significant ones are shown in Figs. 1 to 5.

Figure 1 uses as a basis the order of hardness, from hardest to softest, as obtained with the Rockwell, Alpha scale, which is based on resistance to indentation while under load.

The Tukon-Knoop Method measures the effect of indentation after the load is released. The harder samples, melamine laminate (13), wood flour (14) and mineral-filled phenolics (15) are rated in the same order as by the Alpha Method. In the medium and soft range, represented by polystyrene (11 and 12), methacrylates (1–6), acetate (18), acetate-butyrates (19, 20, 21), nitrate (17), and vinylite (10), the differentiation is poor, due probably to elastic and plastic recovery of the indentation.

Comparing Rockwell Alpha with Rockwell M scale, in which the depth of indentation is measured after removal of the major load we note that wood flour-filled phenolic (14) rates higher than mineral-filled (15), due probably to recovery after removal of the major load.

Materials like polystyrene (11, 12) and vinylite (10), which have a relatively low recovery after indentation are ranked much softer by the M scale than by the Alpha scale. Nylon, nitrate, and laminac laminate (16, 17, 9) behave in the same manner. On the other hand, the methacrylates, (1, 2, 3, 4, 5, 6) CR39 (7) and Laminac clear (8), because of their high recovery after removal of major load, appear to be harder than their position on the Alpha scale would indicate.

Figure 2 shows the rank of the samples as measured by the Rockwell M Scale (PPG-2) and compares it with other M scale methods, with Barcol (PPG), and with Shore Scleroscope.

The M Scale PPG-2 which allows the major load to remain for 7 to 8 sec. after the crank has been tripped and allows 45 sec. of recovery, gives lower readings of hardness on the softer materials than the method PPG-1 in which the major load is removed 1 sec. after the crank comes to rest and reading is made 30 sec. thereafter.

The M Scale readings by the R & H method are considerably lower throughout, since the major load is allowed to remain for 30 sec. The same general order obtains except for the samples of nitron and ethyl cellulose, medium (17 and 23) which are rated higher than would be expected from their ranking by the other M Scale methods.

The Barcol tester uses a pointed indenter, and the load on the indenter decreases in proportion to the depth of indentation. The order of the samples is substantially the same as on the M Scale; exceptions are nylon (16) which tests lower, and Laminac laminate (9) in which the weave in the canvas base retards penetration, resulting in a high reading. This instrument has a limited range since all materials softer than nylon and the acetates gave practically 0 reading.

The Shore Scleroscope measures the degree of instantaneous elastic recovery of an indentation made by impact of a falling tup, regardless of the depth of indentation involved. For this reason, similarly high readings are obtained on materials as widely different in their resistance to indentation as steel and rubber. In the series under consideration, the order by the scleroscope is closely parallel to that of the Rockwell M Scale. The two Saran samples (24 and 25) rank lowest.

Comparison charts were made of the ranking by Alpha Scale and M Scale with Sward hardness, but neither showed any consistent trend nor indication of relationship. Since the Sward value is affected by minor surface irregularities, materials like wood flour and mineral-filled phenolics do not show as high a Sward value as might be expected from their indentation hardness. The highest Sward values were obtained on glass, melamine laminate, and polystyrene injection molded (26, 13, 12), the lowest on the two Sarans and Laminac laminate (24, 25 and 9). The last sample had a rough surface due to the weave in the base fabric.

Bierbaum Scratch Hardness (R & H and L.O.F.) plotted against Rockwell Alpha Scale showed poor correlation, but plotted against M Scale, a better agreement was obtained, as is shown in Fig. 3. Materials which are out of line are Nitron and the two Sarans (17, 24, 25), which test harder than their M Scale ranking. This may be due to a "case hardening" effect or to an elastic recovery of the scratch after the point has passed. Laminac laminate (9) ranks high, probably because of the effect of fabric weave on the surface. There is considerable variation in readings between the two laboratories which may arise from the difficulty of judging the precise edge of the scratch under the microscope.

The Kohinoor value, obtained with lead pencils of different hardnesses is not very informative, depending largely on the operator's judgment of the appearance of a "visible" scratch.

The Moh value ranks all plastics in the range 1 to 3 without much separation.

Test methods utilizing falling abrasive and measurement of optical properties before and after abrasion (haze, gloss) give a ranking which is unrelated to any of the indentation or scratch methods. The Mar Resistance method, A.S.T.M. D 673-44[10] ranks glass (26), CR39 (7), and Laminac (8) in the highest group, and polystyrene, mineral filled phenolic and Saran (24) in the lowest.

Figure 4 shows the rank using as a basis the percentage of original gloss after 400 g. abrasive in the Mar resistance test. The index in the Falling Emery test is increase in haze after abrasion. Many of the samples had considerable haze in the original form, and the assumption that the increase observed was due to the abrasive action may not have been strictly true. The only consistency was in ranking the three best and two worst materials.

The same objection obtains in the measurement of haze on specimens abraded on the Taber Abraser with the CS-10 wheel. On several of the samples the haze after abrasion was lower than the original.

Tests made with the Taber Abraser and CS17 wheels, and determination of weight loss after 200 and 5000 revolutions give a ranking which is unrelated to any of the previous tests. On many of the samples the weight loss for 5000 revolutions was not proportional to the loss at 200 revolutions.

Figure 5 shows weight loss at 200 revolutions plotted against weight loss at 5000 revolutions. Direct proportionality is indicated by the dotted line. It is seen that nylon, most of the methacrylates, melamine laminate, vinylite, and the acetate-butyrates show a fair agreement with this relation. Materials which show a rapid increase in rate of wear are Fibestos, the Laminacs, Nitron, and Saran. Decrease in rate of wear is indicated in CR39, the phenolics, Lucite HC201, ethyl cellulose, and polystyrene (11).

It is of interest that nylon, vinylite, CR39, and the methacrylates, which are among the best in wearing properties, are in the medium range of indentation hardness, while mineral-filled phenolic which is one of the highest in indentation hardness ranks among the poorest in wearing properties.

From the data presented there appear to be at least five different categories of behavior of plastic materials which may be involved in the estimation of the general property called "hardness." They are:

Type	Test
(*a*) Indentation under load..	Rockwell Alpha
(*b*) Indentation after release of load..............	Rockwell M, R, Tukon, Barcol, Bierbaum
(*c*) Instaneous return of the energy of indentation.	Shore Scleroscope
(*d*) Change of optical properties by abrasive wear.	Mar Resistance, Taber Abraser, Falling Emery
(*e*) Loss of weight by abrasive wheel wear........	Taber Abraser

It is evident that "hardness" of plastics, defined as resistance to indentation, is not necessarily a measure of mar, scratch, or wear resistance. It is suggested that the term "hardness" be confined to the concept of resistance to indentation and that other descriptive designations be used to indicate the particular aspect of behavior which is being investigated.

Acknowledgments:

The assistance of the many companies who supplied the plastic materials for test and the various cooperating laboratories in the collection of these data is gratefully acknowledged.

UNIVERSAL PLASTICS TESTING MACHINE[1]*

By G. S. Burr,[2] W. J. Gailus,[3] J. O. Silvey,[4] S. Yurenka,[5] and G. H. Dietz[6]

The specialized requirements of the program of research into the mechanical properties of plastics materials sponsored at the Institute by the Plastics Materials Manufacturers Association—M.I.T. called for a testing machine embodying specialized characteristics. The large range of elastic and plastic properties of plastics materials and their sensitivity to time rates as well as to ambient atmospheric conditions made it necessary to develop testing equipment having unusual speeds, travel, and controls. The principal requirements set up for the machine finally designed were the following:

Capacity, 30,000 lb.
Rates of crosshead motion, 0.005 to 30 in. per min., in continuously variable increments.
Crosshead motion, 18 in.
Minimum inertia in the weighing and recording mechanism.
Positive control over crosshead motion in either direction with fastest possible reversal in direction.
Reversal of load on specimen without backlash when passing through zero, with grips capable of applying loads in tension and compression continuously on the same specimen.
Controls for:
- (*a*) Constant rate of crosshead motion.
- (*b*) Constant rate of load application.
- (*c*) Constant rate of strain.

For possible future consideration: constant rate of stress. Size sufficient to allow the incorporation of temperature-control boxes.

Description of Machine

A brief description of the machine (Fig. 1) and its controls follows:

In view of the special requirements imposed upon this machine, it was necessary to design it with some unusual specifications in mind. The load weighing system should have a relatively high speed of response, in other words, low inertia; it should be capable of recording an extremely wide range of full-scale loads, from a few pounds to 30,000 lb., and should suffer negligible deflection under the action of the load. These requirements were met with a weighbar design utilizing bonded-wire strain gages and a sensitive amplifying and recording system. Inasmuch as it was also desired to measure creep and relaxation properties, the load recording system needed to have good stability over reasonable lengths of time.

These requirements dictated the

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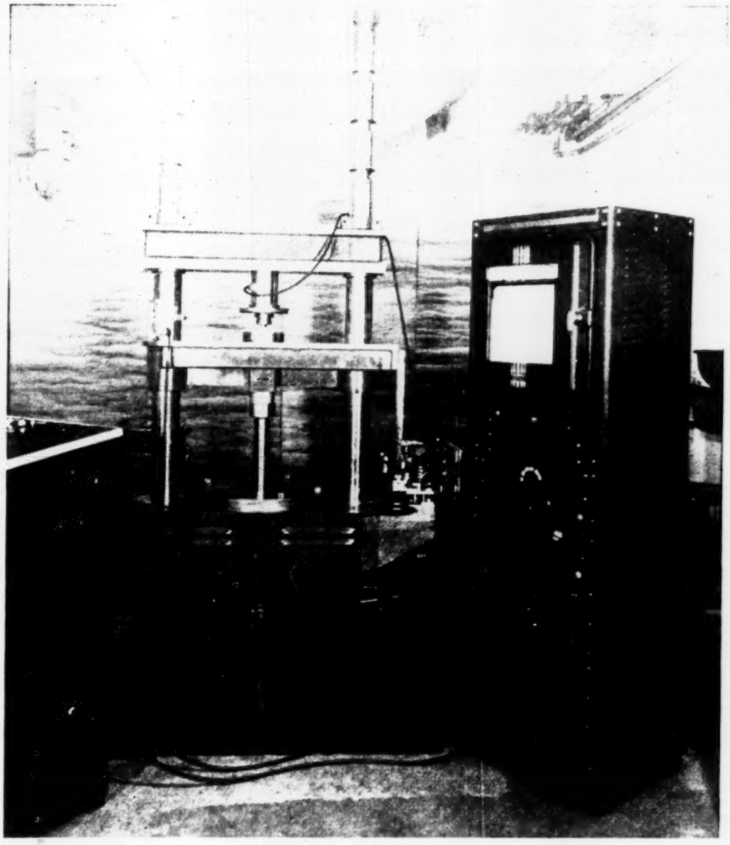

FIG. 1.—Universal Plastics Testing Machine.

use of a servo-mechanism type of control, operating through electrical, mechanical, and hydraulic circuits to move a double-ended hydraulic piston.

A simplified block diagram of the system is shown in Fig. 2, illustrating the manner in which the necessary flexibility of operation is achieved by feeding the desired signals into either the controlling servo or the recorder. Because the chart of the recorder is moving at a constant rate, the time axis on the chart represents the particular function which is under the control of the servo network. Thus, load can be recorded against a constant rate of strain or jaw separation; sample extension or jaw separation can be recorded against a constant rate of load, and so on, in any desired combination.

Inasmuch as it is very often desired to use the movement of the moving crosshead as a measure of deformation, particularly in compression tests, the machine was designed so as to reduce deflection of the structural members and the crossheads to a minimum when under full load.

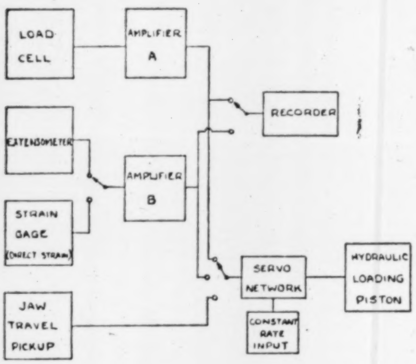

FIG. 2.—Block Diagram of Testing Machine System Showing Relationship of Hydraulic System, Servo Controls, Weighing System, and Recorder.

Body:

The body of the machine shown in Fig. 1 is designed to be light and trim but sturdy, affording the flexibility required for the plastics test program. With proper jigs and fixtures it can be used for tension, compression, bending, shear, some types of fatigue tests, and high velocity creep or low velocity impact. The distance between the crossheads is completely variable between 6 and 53 in. and the movable crosshead, driven by a double-ended hydraulically operated piston, has a total up or down travel of 18 in. The basic frame has been designed to deflect not more than 0.002 in. under a maximum load of 30,000 lb. This deflection occurs only under extreme conditions, that is, when the maximum load is applied with the columns in tension and the upper crosshead at the uppermost position. The deflections under ordinary conditions are less. Stresses set up in the frame by the 30,000-lb. maximum load are relatively small.

Welded construction is utilized throughout the machine to achieve minimum weight with maximum rigidity, especially in the moving and fixed crossheads. Welded ribs and box sections give a large strength-to-weight ratio to these parts in addition to making their construction possible from standard rolled plates, bars, and tubes.

For accurate leveling, the machine is supported at three points by a welded rectangular "picture" frame made of 4 by 4 by $\frac{1}{2}$-in. angles and channels. The "picture frame" itself rests on four shock mounts secured to the foundation with four $\frac{1}{2}$-in. studs. Because the height of the machine exceeds the floor-to-ceiling distance within the laboratory assigned to the project, the frame and lower half of the base of the machine are contained in a shallow pit. This brings the normal working space within the machine to a convenient level for the operator.

A Hanna H.P. 16 double-ended hydraulic cylinder drives the movable crosshead by means of a $2\frac{1}{2}$-in. diameter piston rod. The cylinder is accurately scrape-fitted and is secured to the machine by eight 1-in. bolts in such a way that it may be disassembled for repairs or inspection if necessary.

The upper end of the piston rod is threaded into the lower, or movable crosshead. At the center of the upper surface of this crosshead a $1\frac{1}{2}$-in. 12 NC thread is incorporated to provide a means for the attachment of various jigs and fixtures.

The movable crosshead is guided by 4-in. diameter precision ground cylindrical guide columns. To reduce friction, and to increase accuracy of motion, 7-in. long bronze bushings serve as the crosshead

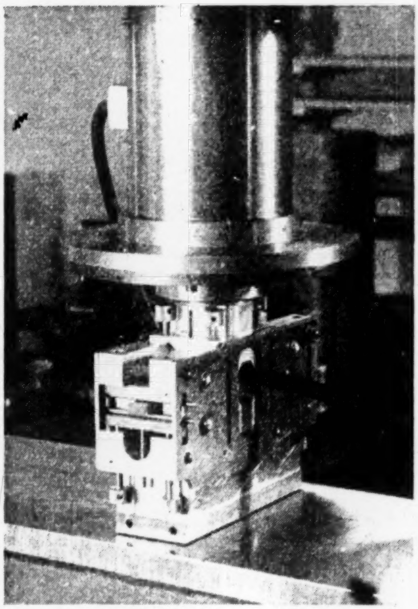

FIG. 3.—General View of Load Cell Attached to Upper Crosshead of Machine.

guiding surfaces. The guide columns provide the solution to the problem of correct alignment and straight travel of the crossheads. The straightness of the columns (as guaranteed by the manufacturer) is within 0.002 in. in 9 ft. The columns are parallel to within the same tolerance. Six circumferential rectangular grooves in the upper part of the columns are provided to fix the upper crosshead at different levels. A split ring and double-wedge arrangement is employed to lock the upper crosshead rigidly to the columns, permitting no detectable movement in either direction under all loading conditions. The lower ends of the columns are secured to the base of the machine which also contains the cylinder. The principal components of the base are two 3-ft. long sections of 8-in. extra strong pipe reinforced by several $\frac{3}{4}$-in. stiffeners and sandwiched between two $1\frac{1}{2}$-in. thick steel plates.

Load Indicator:

Attachable to either crosshead is the load indicating device, or load cell. It consists essentially of a special weighbar to which is bonded a resistance wire strain gage. Axial strains in the bar are measured and, being proportional to the load, are calibrated in pounds. The first cell built was designed for an operating range up to 5000 lb. and is typical in its details of the load cells to be employed for other ranges (see Figs. 3 and 4). It has five main components: a diaphragm, flange, tube, weighbar, and attaching bolt. All of the parts with the exception of the weighbar and diaphragm are made of mild steel. The weighbar consists of a cylinder of No. 4140 nickel-alloy steel (in a hardened and drawn condition—yield point of about 110,000 psi.) machined and ground at one end to form a "weighing tube" with a mean diameter of 0.860 in. and a wall thickness of 0.030 in. The other end is threaded for testing grip attachment.

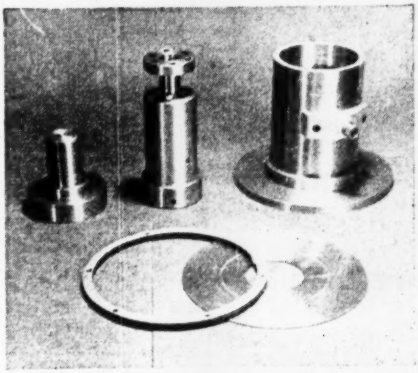

FIG. 4.—Disassembled View of Load Cell Showing Diaphragm, Flange, Tube, and Weigh bar.

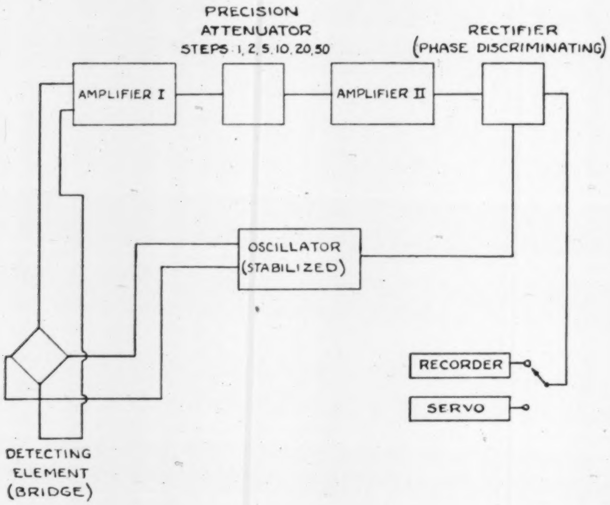
PRECISION ATTENUATOR STEPS 1, 2, 5, 10, 20, 50
RECTIFIER (PHASE DISCRIMINATING)
AMPLIFIER I
AMPLIFIER II
OSCILLATOR (STABILIZED)
RECORDER
SERVO
DETECTING ELEMENT (BRIDGE)

FIG. 5.—Block Diagram of Load and Extension Recording Circuits.

The weighbar is designed to have a maximum deflection of 0.002 in. under the 5000-lb. load. This corresponds to a stress in the weighing tube of about 60,000 psi. (tension or compression).

The steel diaphragm 0.0625 in. thick (designed to take up just 1 per cent of the axial load throughout the range of the load cell) is provided to prevent nonaxial loading of the weighing tube. Another of its important functions is to prevent torque from being transmitted to the weighing tube in attaching a testing grip to the load cell.

Load and Extension Recording Circuits:

The bonded wire strain gage is the principal element in the load cells and is one of the principal methods of detecting direct strain and small extensions. Therefore, the amplifying circuits were designed primarily to be used with this device, but are adaptable to other types of detecting elements that may be necessary in the development of adequate extensometers.

The circuit, shown in block form in Fig. 5, is based on the familiar a-c. Bridge principle, with certain refinements to make it suitable for the present application. The load cells are designed to cover a 50 to 1 full-scale range, and the minimum full-scale strain that is to be amplified is of the order of 30 microinches per inch. This requires large amplification and special care to eliminate the extraneous noise signals that appear in low level circuits. A special rectifier circuit was developed to provide an output that was a true measure of the resistance unbalance alone of the bridge, excluding capacity effects and stray hum pickup. Because the recorder operates on the deflection principle, rather than null balance, special care was necessary to insure that oscillation output be constant in spite of line voltage variations and other disturbances.

A precision attenuator was incorporated in the load amplifier to provide a means of varying the full-scale range of the recorder in the following discrete steps: 1, 2, 5, 10, 20, and 50 lb. These values are multiplied by a factor depending on the range of the particular load cell in use. In this manner, each cell needs to be calibrated only at the lowest value in its range, usually by dead weights, and the calibration can then be considered effective over its entire 50:1 range.

The recorder used is a high-speed ink-recording Leeds & Northrup Speedomax, with a specially built chart drive to provide chart speeds ranging from 2 to 50 in. per minute. The recorder will sweep a full-scale deflection in about 1 second, and will faithfully reproduce smaller deflections in proportionally less time. The chart width is 10 in. By means of zeroing controls on the amplifier it is possible to position the pen at center-scale or at any other convenient point in order to measure loads in either direction.

A separate circuit provides a small pip, or disturbance, on the record whenever the movement of the pulling jaw is stopped or started. This facilitates the interpretation of data for creep or relaxation studies. An overload circuit operates to stop the machine whenever the load exceeds a safe preset value thus acting to prevent damage to the load cell.

Servo Controls:

The controller design was complicated because of the many modes of operation required, the relatively large ranges through which the

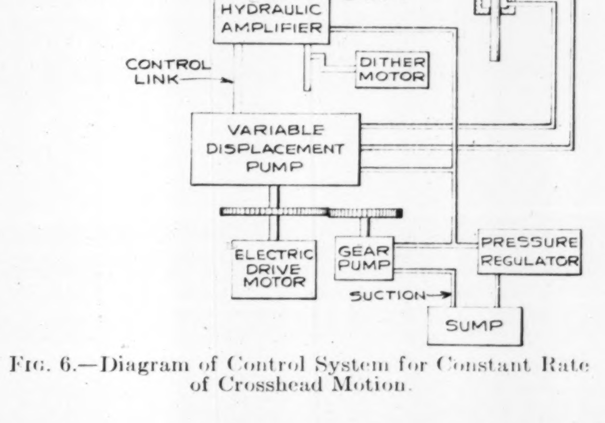
WEIGHING HEAD
SAMPLE
VARIABLE SPEED DRIVE
REVERSING MAGNETIC CLUTCHES
CONSTANT SPEED MOTOR
RACK
ERROR SHAFT
HYDRAULIC AMPLIFIER
CONTROL LINK
DITHER MOTOR
VARIABLE DISPLACEMENT PUMP
ELECTRIC DRIVE MOTOR
GEAR PUMP
PRESSURE REGULATOR
SUCTION
SUMP

FIG. 6.—Diagram of Control System for Constant Rate of Crosshead Motion.

rates and the magnitude of load were required to be varied, and because of the accuracy with which the rates of load, crosshead motion, and strain were to be controlled. To accomplish all objectives, it was decided to control the movable crosshead by means of a servomechanism. The mechanics of changing from one mode of operation to another obviously had to be kept as simple as possible.

Constant Rate of Crosshead Motion.—The control system for constant rate of crosshead motion is shown schematically in Fig. 6. The servomotor is the hydraulic piston of the testing machine. This piston is controlled by means of a variable-displacement hydraulic pump. The pump stroke is controlled by means of a hydraulic amplifier consisting of a pilot valve, a control piston to control the pump stroke, and feedback linkages to make the control piston motion proportional to pilot valve motion. The sleeve in which the pilot valve moves is constantly dithered by means of an eccentric driven by a small auxiliary electric motor to keep friction between the pilot valve and sleeve at a minimum. Oil under pressure to supply the control piston is provided by a small gear pump which is driven by the same electric motor that drives the variable displacement pump. This gear pump also makes up any leakage that may occur in the variable displacement pump and the large piston of the testing machine. Control pressure is maintained approximately constant by means of a small ball-spring type regulator which returns all excess oil to the sump.

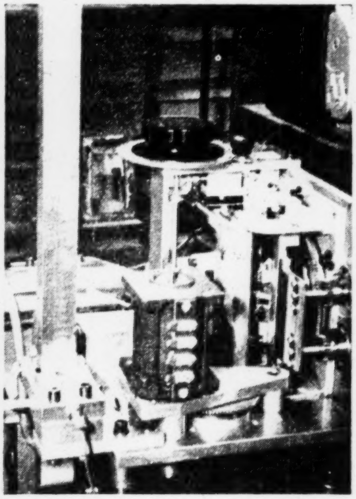

FIG. 7.—Detail of Servo System Showing Rack Attached to Movable Crosshead, Ball and Disk, Differential Cage, and Input Motor.

In constant rate of crosshead motion the desired input motion is imparted to one side of a mechanical differential. The other side of the differential is driven by a rack fastened to the crosshead of the testing machine. The differential cage is connected through gears and linkages to the pilot valve. The directions of motion are such that if the position of the crosshead does not correspond to the position indicated by the input, the hydraulic amplifier will displace the pump, and the crosshead will move in the proper direction to reduce the difference between required and actual crosshead position to zero. Hence, to produce constant rate of crosshead motion, it is only necessary to rotate the input gear of the differential at a constant speed. This is accomplished by driving the input gear by means of a continuously variable-speed drive of the ball-disk integrator type and a small constant-speed electric motor. Various speed ranges are obtained by means of change gears. A pair of magnetic clutches is used to obtain quick reversal of motion (see Fig. 7).

An auxiliary device is located in the hydraulic amplifier which prevents a positional lag which would otherwise occur when the crosshead is moving at a constant rate.

Constant Rate of Load.—The system used for load control is shown schematically in Fig. 8. To understand its operation, let us first ignore the presence of the derivative network and assume that amplifier No. 2 is connected directly to amplifier No. 3. In this form of control the controlling quantity is load rather than crosshead position. (In Fig. 8, the circuit from vacuum tube amplifier No. 1 runs to the load cell.) Consequently, the amplified electrical signal from the load cell is compared with the input signal. The input signal is obtained from a potentiometer, and the difference between input and output signals is obtained by connecting them in series with the polarities reversed. The difference, or error, voltage is amplified by amplifier No. 2 and amplifier No. 3 until it is of sufficient power to operate the electric control motor. This control motor is geared to the input gear of the differential. The other gears of the differential are meshed with the same gears as they were in constant rate of crosshead motion operation. The connections between the hydraulic amplifier, hydraulic pump, main piston, and feedback rack are exactly as they were for constant rate of motion. Consequently, the operation of the system is as follows: If the output load is different from that called for by the input signal, an error signal enters amplifier No. 2, passes to amplifier No. 3, and results in rotation of the electric control motor. This rotation changes the input to the differential, causing motion of the pilot valve, and hence motion of the main piston. A change of piston position, however, alters the load applied to the sample, thus changing the output of amplifier No. 1 until the output signal is equal to the input signal.

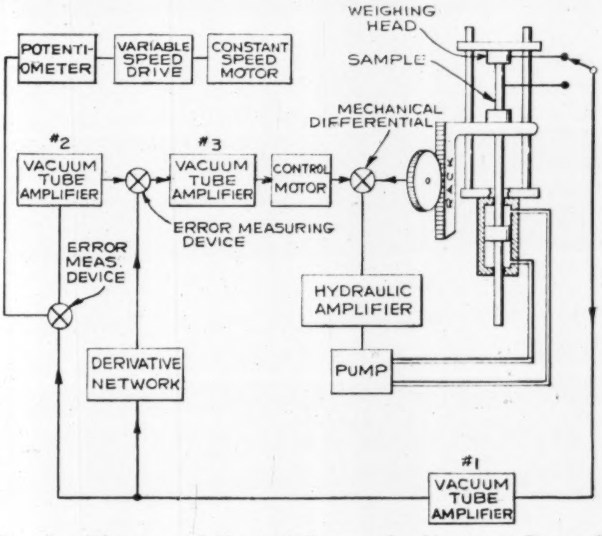
WEIGHING HEAD
SAMPLE
POTENTIOMETER
VARIABLE SPEED DRIVE
CONSTANT SPEED MOTOR
MECHANICAL DIFFERENTIAL
#2
VACUUM TUBE AMPLIFIER
#3
VACUUM TUBE AMPLIFIER
CONTROL MOTOR
RACK
ERROR MEASURING DEVICE
ERROR MEAS. DEVICE
HYDRAULIC AMPLIFIER
DERIVATIVE NETWORK
PUMP
#1
VACUUM TUBE AMPLIFIER

FIG. 8.—Diagram of Control System for Constant Rate of Load (Adaptable to Constant Rate of Strain).

The electrical mixing circuit as well as the derivative network is used to produce greater stability of the system by introducing some derivative or "anticipation" into the control circuit. This enables the machine to test materials with a large range of properties without adjusting the constants of the vacuum-tube amplifiers.

Constant rate of change of load can be applied to the sample by rotating the shaft of the linear input potentiometer at a constant rate of change.

Constant Rate of Strain.—If the circuit in Fig. 8 is run from amplifier No. 1 to the extensometer on the sample and the motion of the extensometer is converted into a change in voltage (by change in resistance, change in inductance, etc.), the servo controls employed for constant rate of load can be employed for constant rate of strain. The only difference in the operation of the servo controls is that the signal comes from an extensometer attached to the specimen instead of from the load cell. The operation of the various amplifiers and the rotation of the linear input potentiometer are unchanged.

Advantages of these systems of control are: once the transients have died out they are quite accurate; once calibrated, the settings for particular rates do not change; forces and pressures controlled directly by the calibrated speed drives are quite low. One minor disadvantage of servo control systems is that because they are actuated by an error between the input and the controlled quantity, they cannot begin acting until after an error has been produced. Hence, when a sudden input or rate of input signal is applied, a certain amount of time is required before the error settles again to zero. However, this time interval can be reduced to a value of approximately 1 sec. in the case of this machine.

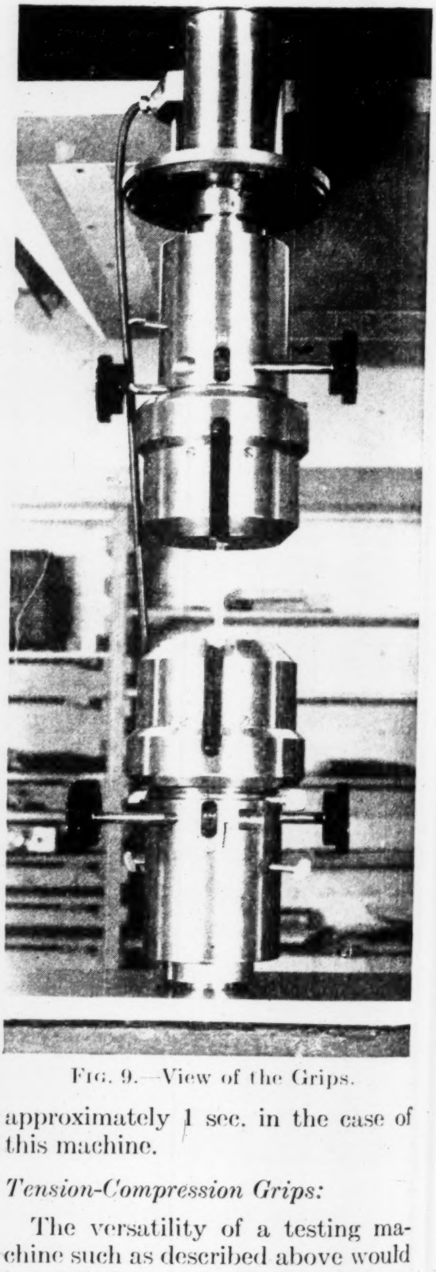

Fig. 9.—View of the Grips.

Tension-Compression Grips:

The versatility of a testing machine such as described above would be of little practical value without having a method of applying to it the now fairly standardized procedures employed in the testing field.

Thus came about the development of a different type of specimen grip which is described in detail below.

A photograph of the grips is shown in Fig. 9. The grips are of the open front type, that is, a specimen may be inserted directly from the side—a very desirable feature as it insures correct alignment of the specimens with ease and rapidity, which is especially important when a large number of tests have to be run.

Each grip is freely suspended by means of a special yoke in such a way that the grip floats on a $\frac{3}{4}$-in. polished steel sphere, thus insuring automatic alignment of the grips in tension testing. The clearances provided within the grips allow vertical freedom within a solid angle of 10 deg. Although the lower grip is in an inverted position and does not bear upon the steel sphere when not in use, other spherical surfaces support the weight of the jaw and keep it in an upright position until the slack is taken up by the specimen.

The special feature of these grips comes in the fact that once aligned they may be locked in position and the specimen subjected to compression as well as tension up to the maximum design load, that is, 30,000 lb. Adjustable wedges are provided which can be opened to accommodate a specimen with a maximum width of $1\frac{1}{4}$ in. and a thickness up to $\frac{9}{16}$ in. In operation, a specimen is inserted between the wedges and clamped manually with any reasonable degree of force, after which the wedges continue to tighten on the specimen automatically, with a force proportional to the tension in the specimen. This force can be "locked" onto the specimen at any desired level so that the specimen may then be subjected to a compressive load. After the specimen is broken, the ends can be easily released by the same manual control which was used to secure it initially.

To insure positive gripping of specimens of different hardness, several sets of removable hardened face plates are provided with the wedges. These have graduated degrees of sharpness and tooth depth. Other than flat specimens can be accommodated by using specially formed face plates.

The satisfaction of the specification of a 30,000-lb. rated capacity for the grips and the inclusion of the special features desired resulted in grips weighing about 60 lb. each. However, the size of the grips is held to a minimum by such close packing of the component parts that the grips actually weigh about 90 per cent as much as a solid piece of steel having the same outside dimensions. The over-all height of each grip is 11 in. and is designed to obtain the maximum capacity of specimen length between the crossheads of the testing machine.

Acknowledgments:

The authors wish especially to acknowledge the assistance of Leon Marsh, consulting engineer, in the design of the body of the machine; G. S. Brown of the Massachusetts Institute of Technology Servo-mechanisms Laboratory for suggestions respecting the controls; L. J. Lipschutz for assistance in the design of the load cell; W. H. Campbell for assistance in the development and assembly of the various components; and A. C. Ruge in the development of the weighbar.

DISCUSSION

Mr. E. M. Redstreake.[1]—Is the load indicated on a dial?

Mr. A. G. H. Dietz (*author*).—The load is indicated on a moving paper chart to provide a pen and ink record of every test.

Mr. Clare L. Milton, Jr.[2]—What is the frequency of the oscillator in the load-measuring circuit?

Mr. Dietz.—The oscillator frequency is 400 cycles per second.

Mr. L. N. Phillips.[3]—One of the great virtues of this apparatus is the ability to go continuously from compression to tension without having to reset the grips or extensometers in any way. It should therefore be possible to get complete hysteresis diagrams. There are two possible sources of error here that I should like to query. First: Is there absolutely no backlash from the rack and pinion mechanism used to record the motion of the crosshead? Second: What shape of test specimen has been decided upon that gives correct readings in both tension and compression tests?

Mr. Dietz.—Backlash has not been evident. We were somewhat concerned about this, but performance has been satisfactory.

As for the proper type of specimen to use for tension and compression, we are still experimenting. The specimen will have to be short, evidently, to avoid buckling. At the same time stress concentrations around the fillets near the jaws are to be avoided and, frankly, that has not yet been completely solved. So far, our work is very rudimentary (the grips were just finished about two weeks ago) but a specimen length between jaws of about five to six times the least cross-sectional dimension has seemed satisfactory. More work needs to be done on this problem.

Mr. Lucius Gilman.[4]—I do not see why you have to have two servo devices in the circuits where you are operating, for example, at a constant rate of loading.

Since there are two counter voltages coming in at the same point, why can't the differential voltage which comes out be used to control the speed of the drive motor instead of having to feed it through the mechanical servo?

Mr. Dietz.—This could be done, but it would require that the hydraulic piston be replaced by an electric motor of the same power and that the power amplification of the mechanical servo (which is approximately from $\frac{1}{100}$ to 2 hp.) be obtained by electrical means. The use of the existing motor which drives the hydraulic pump is impossible because of undesirable characteristics as a control motor.

Having decided that the variable

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[1] Tinius Olsen Testing Machine Co., Philadelphia, Pa.

[2] Chief, Plastics Development Branch, Army Prosthetics Research Lab., Washington, D. C.

[3] British Commonwealth Scientific Office, Washington, D. C.

[4] Chief, Plastics Lab., Picatinny Arsenal, Dover, N. J.

displacement pump-hydraulic piston combination would be the simplest system to use for constant rate of crosshead motion, the easiest way to obtain constant rate of load was by means of the two-servo system.

MR. J. R. STOVALL.[5]—I should like to ask two or three questions. Did you have any trouble when you used your rack and gear arrangement down to the low speed, which I think was 0.01 in. per min.? That is rather a low speed for using a rack and gear.

MR. DIETZ.—It does seem like a rather slow motion but it seems to be operating well. There is some spring loading to remove backlash, and the rack is accurately cut. As a matter of fact it has been used at speeds as low as 0.003 in. per min. with no detectable error using the same gear differential. It was checked against dial gages placed directly under the crosshead to check the motion of the dial gages against the motion recorded on the chart and there was no appreciable error. "Appreciable" needs definition. The requirement was that the system should operate with not more than 1 per cent error. The motion of the crosshead was within this requirement.

MR. STOVALL.—The second question is this: Judging from the diagrams, I believe the load indications were made by recording voltage output. Then you would depend on a linear constant gain amplifier? Is that correct?

MR. DIETZ.—Yes. The amplifier is a linear constant gain circuit.

MR. STOVALL.—Then the third question is this: Also from the diagram, I believe the ratio on the attenuator was 1 to 50.

MR. DIETZ.—That is correct.

MR. STOVALL.—And you use a single cell for that?

MR. DIETZ.—Yes.

MR. STOVALL.—The reason I ask is that, if you use a single 5000-lb. cell, you have a 50 to 1 ratio. Assuming that you have a 0.1 per cent sensitivity, that gives you a dynamic range for the strain gage of 50,000 to 1. Now were you able to work the strain gages at that dynamic range? That is quite a large range. I have never seen them used in that wide a range before.

MR. DIETZ.—Yes, we have used that load cell mostly so far on the 100-lb. range—that is to say, the 50 to 1 range. The cell has been checked a number of times against dead weights and also has been checked for the full 5000-lb. range against the Morehouse ring, and there has been not more than 1 per cent error in the record.

MR. H. M. QUACKENBOS, JR.[6]—I have three questions about this remarkable piece of equipment.

1. Whereas the electrical system would be very quick to respond, would not the pump be rather slow?
2. On the 100-lb. range the maximum strain gage reading is 30 microinches per in. A precision of ±1 per cent therefore corresponds to only $\frac{1}{3}$ microinch. Could not a small temperature differential within the system cause thermal expansion greater than $\frac{1}{3}$ microinch and so invalidate the precision?
3. How often will recalibration be necessary to check the constancy of the electric strain gages and thus the constancy of the weighing system?

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[5] Electronics Department, Tinius Olsen Testing Machine Co., Philadelphia, Pa.

[6] Bakelite Corporation, Bloomfield, N. J.

MR. DIETZ.—The pump is a Vickers Variable Displacement Pump, and the total motion of the control valve for full speed in one direction—full pressure in one direction and full pressure in the other direction—is about $\frac{1}{8}$ in. It can be changed from full speed forward to full speed back in a fraction of a second.

There is a very small overrun which is unavoidable, but it is very small. It is less than the requirement of not more than 1 per cent error which was established for the system.

The possibility of temperature errors has been borne in mind. The system—the machine itself—is in an air-conditioned space in which the temperature and humidity conditions are kept reasonably constant. We try to maintain the A.S.T.M. standard conditions. The gages on the weighbar are within the heavy enclosing cell and the temperature within it probably undergoes a very small change. If there were marked temperature changes within the system, they might easily throw it off more than 1 per cent.

For the time being, we expect to calibrate frequently against a Morehouse ring or against dead weights until we find out more about the stability of the system.

The previous history of strain gages in other pieces of equipment in the Institute has been quite satisfactory, especially in air-conditioned space. The Textiles Laboratory has a history going back over perhaps two years. It has been found that when gages are baked onto the surface—not just put on with Duco or similar types of cements—and when they are also very carefully sealed against any moisture change by a heavy coating of wax, paraffin, or something of that nature, the gages have had a good history of stability.

It is recognized that stability is certainly something that must be watched, but we felt that in a piece of research equipment it was something that could be checked at rather frequent intervals.

MR. MILTON.—While there is little doubt that strain gages are capable of being employed over the 50,000 to 1 range that you have used, to take advantage of this range requires the measurement of voltages to fractions of a microvolt or balancing to a similar precision. Is that not correct?

MR. DIETZ.—That is correct.

MR. MILTON.—The difficulties in designing a recording apparatus of this precision are well known and Mr. Dietz and his colleagues deserve congratulation. The familiar method of eliminating the intrusive voltages (which must be considerably larger than your precision of measurement) by the use of a rapid reversing switch like that of the Wenner potentiometer would be most difficult of application to a recorder. Would you care to go into any detail as to how you took care of intrusives in the gages and a-c. amplifier which the block diagram indicated as the first stage of your apparatus?

MR. DIETZ.—It is true that stray hum pickup in the circuit can be several times the magnitude of the signal. It is kept to a minimum by very careful construction. The residual is isolated from the recorder by means of the phase discriminator circuit.

If we are going to operate at loads much less than 100 lb., we do not intend to use that 5000-lb. cell. A lighter load cell will be built, since we have no expectation that we will be able to go to much less than 100 lb. with a high degree of accuracy with this cell. We expect probably to have three cells: one for low ranges up to 500-lb. maximum, one for 5000-lb. maximum, and a third for the full capacity of the machine.

MR. J. J. LAMB.[7]—You mentioned in several cases a variable rate. Do you mean that you can feed a signal in—such as a sine wave—to give you a variable rate of straining?

MR. DIETZ.—To a certain extent you could. For instance, suppose that in the rate of load mechanism instead of rotating the potentiometer at a constant rate it were rotated at a variable rate, such as one that would correspond to a sine wave. There is no reason why the machine should not respond to that type of input as well as a constant rate input.

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[7] Physicist, National Bureau of Standards Washington, D. C.

TESTS FOR THERMAL DIFFUSIVITY OF GRANULAR MATERIALS*

By William L. Shannon[1] and Winthrop A. Wells[1]

Synopsis

This paper presents the procedures, analyses, and results of tests to determine, using a simplified test procedure, the thermal diffusivity of several granular materials of types frequently used for base-course construction of highways and airports. Determinations were made with the materials in the frozen and unfrozen condition at several different unit weights and water contents. From the test data obtained, the volumetric heat capacity and the thermal conductivity for each thermal diffusivity determination were computed. The method, where used to determine the thermal conductivity, is relatively simple and inexpensive in comparison with the more precise methods generally used, yet results of tests on one material using the simplified method and a precise method compare favorably.

In connection with an investigation of methods for the design of airport pavements from the standpoint of frost action, the New England Division Office of the Corps of Engineers has conducted a research study of the thermal diffusivity of several typical types of materials used for airfield pavement and base construction. The purpose of this study was primarily to provide numerical values for the thermal properties of the typical materials tested, which values were desired for analysis of frost-penetration data obtained by observation and computed by formulas.

Several investigators, principally Patten (**1**),[2] Shanklin (**2**), Kennelly and Shepard (**3**), Teichmuller and Humen (**4**), Ingersoll and Koepp (**5**), and Berggren (**6**), have reported results of tests to determine the thermal properties of soils. Of those listed, only Berggren reported results of tests upon soils in the frozen state. The unit weight at which the soil was tested was reported in only two of the investigations; those by Patten and Ingersoll and Koepp. All except Berggren reported the water content of the soil for each test. The Bureau of Reclamation (**7**), in connection with the Hoover Dam project, reported a very complete series of tests to determine the thermal properties of concrete and component aggregates.

A review of these data indicates that the thermal properties of soils are dependent upon their water content, component parts, unit weight, and soil condition (frozen or unfrozen). These data also indicate in general that testing is the only method for obtaining satisfactory numerical values for the thermal conductivity of soils. The volumetric heat capacity may be computed satisfactorily for most soils from values for the unit weight and water content of soil and the specific heat of water, ice, and soil.

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[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1054.

The method used for determining the thermal diffusivity was chosen primarily for its simplicity of apparatus and test procedure. Essentially the method consists of computing the thermal diffusivity from observed temperature changes at the midpoint of a cylinder due to a sudden change in the temperature of the exterior of the cylinder.

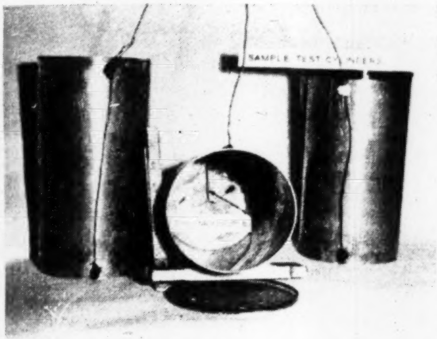

FIG. 1.—Cylinders Used for Thermal Diffusivity Tests.

DESCRIPTION OF APPARATUS

The apparatus used for these tests consists of the following:

Cylinders.—A brass cylinder of height equal to twice the diameter with brass top and bottom covers, as shown in Fig. 1.

Constant-Temperature Bath.—A constant-temperature bath in which the brass cylinders may be totally immersed, as shown in Fig. 2.

Thermocouples.—Thermocouples with Leeds & Northrup potentiometer to measure temperature at the center of the cylindrical sample, as shown in Fig. 1.

Compaction Equipment.—Compaction equipment to obtain uniform density throughout the sample. The compaction equipment used was that developed by Dr. Liang-Sheng Chen of Harvard University.

Freezing Cabinet.—A freezing cabinet to freeze the samples to a low temperature (approximately 0 F.). This cabinet was located in a cold room whose temperature was controlled at approximately 40 F.

MATERIALS TESTED

The following materials were tested. The gradation and specific gravity of all the materials are shown in Fig. 3 and Table I, respectively:

Lowell sand consisted of a cohesionless, siliceous sand from a glacial outwash deposit at South Lowell, Mass.

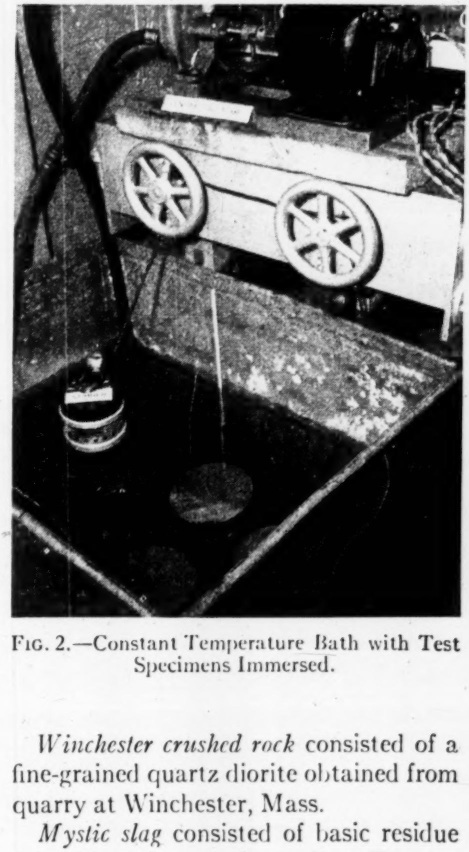

FIG. 2.—Constant Temperature Bath with Test Specimens Immersed.

Winchester crushed rock consisted of a fine-grained quartz diorite obtained from quarry at Winchester, Mass.

Mystic slag consisted of basic residue from blast furnace located at Everett, Mass.

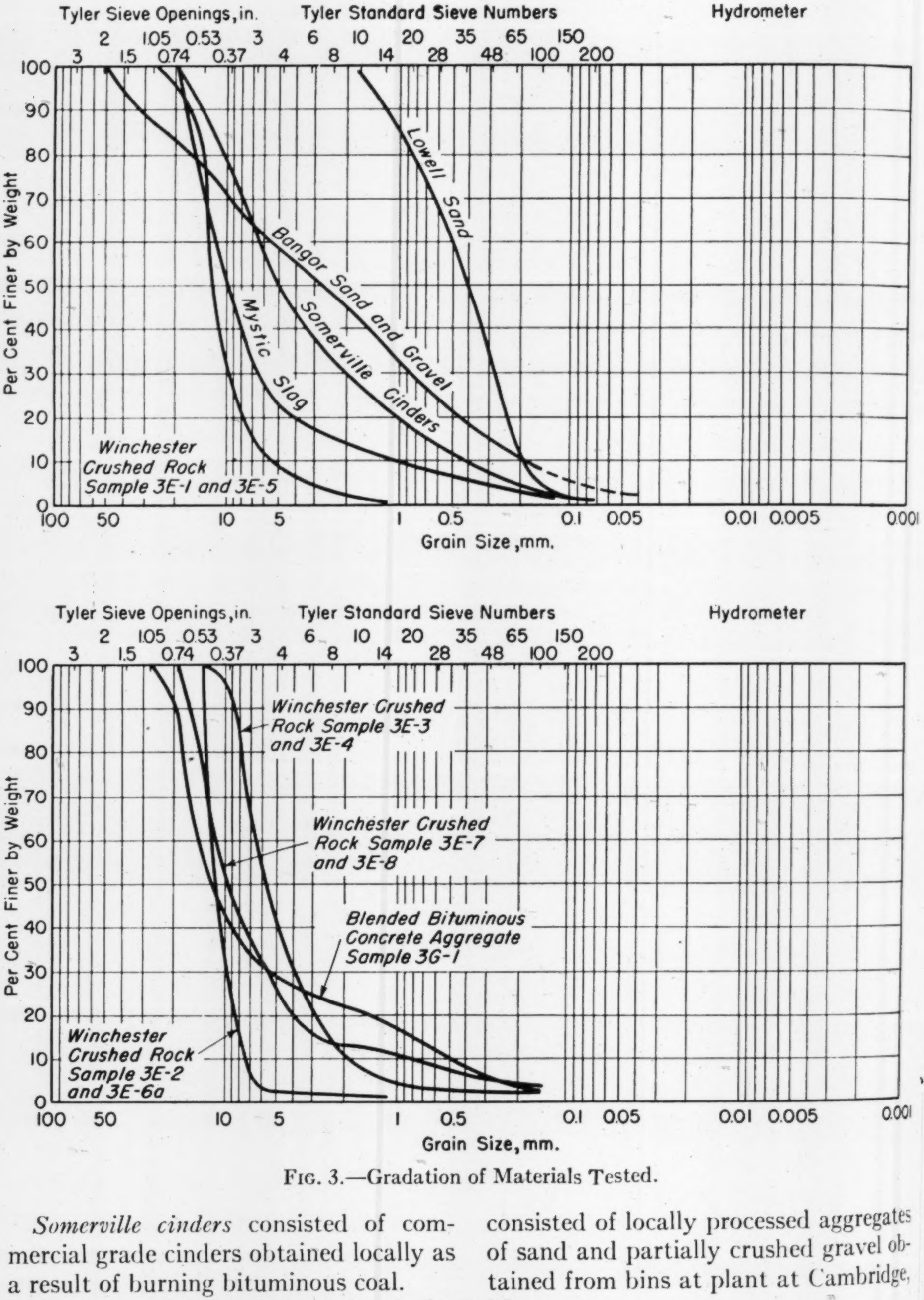

FIG. 3.—Gradation of Materials Tested.

Somerville cinders consisted of commercial grade cinders obtained locally as a result of burning bituminous coal.

Bangor sand and gravel consisted of a well-graded sand and gravel of glacial origin obtained from Bangor, Maine.

Blended bituminous concrete aggregate consisted of locally processed aggregates of sand and partially crushed gravel obtained from bins at plant at Cambridge, Mass.

Asphaltic bituminous concrete consisted of the blended bituminous concrete aggregate and 4.5 per cent bitumen.

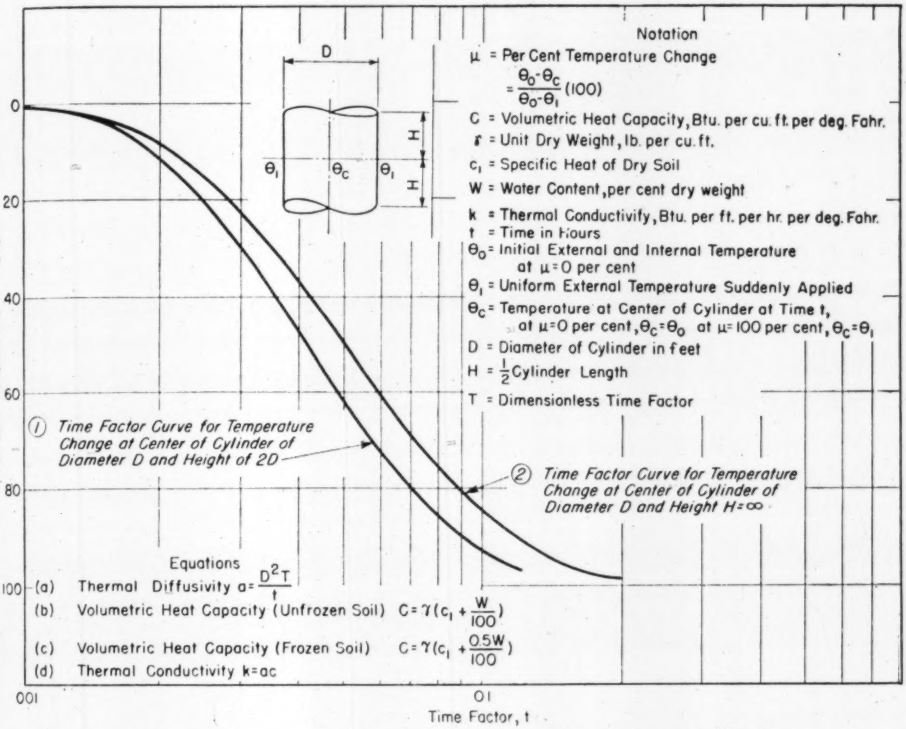

FIG. 4.—Time Factor Curves for Temperature Change at Center of a Cylinder.

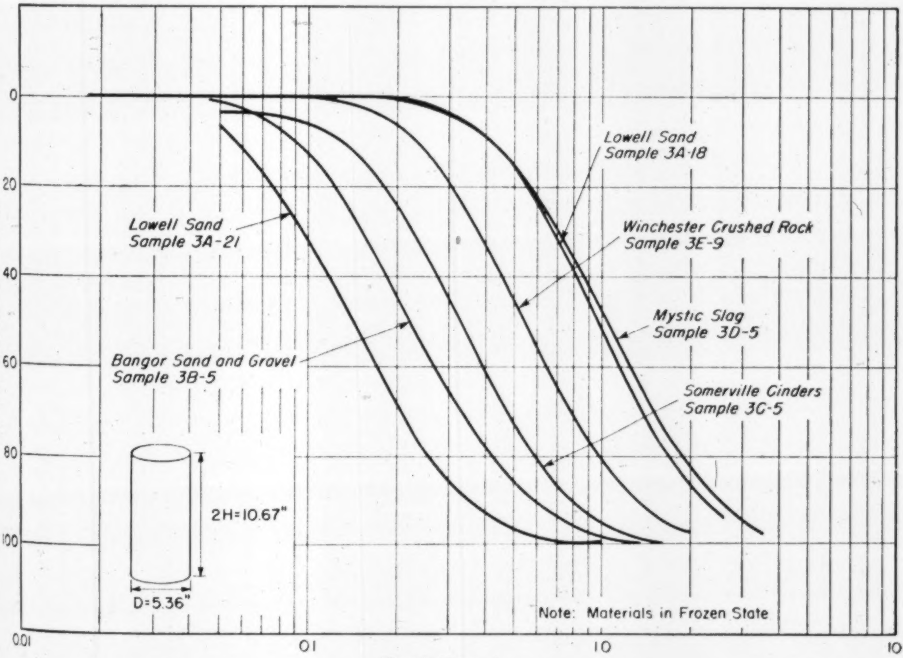

FIG. 5.—Typical Time Curves for Thermal Conductivity Determinations.

TABLE I.—SUMMARY OF DATA.

Materia	Specific Gravity	Specific Heat of Dry Soil[a]	Unfrozen							Frozen						
			Laboratory Sample	Unit Weight, lb. per cu. ft.	Unit Dry Weight, lb. per cu. ft.	Water Content, w, per cent dry weight	Volumetric Heat Capacity C, Btu. per cu. ft. per deg. Fahr.	Thermal Diffusivity, a, sq. ft. per hr.	Thermal Conductivity, k, Btu. per ft. per hr. per deg. Fahr.	Laboratory Sample	Unit Weight, lb. per cu. ft.	Unit Dry Weight, lb. per cu. ft.	Water Content, w, per cent dry weight	Volumetric Heat Capacity, C, Btu. per cu. ft. per deg. Fahr.	Thermal Diffusivity, a, sq. ft. per hr.	Thermal Conductivity, k, Btu. per ft. per hr. per deg. Fahr.
Lowell Sand (Well graded medium to course sand)[b]	2.66	0.20	No. 3A-4	105.2	105.0	0.2	21.2	0.00887	0.188	No. 3A-18	106.2	106.2	0.17	21.3	0.00869	0.185
			No. 3A-4a	105.2	105.0	0.2	21.2	0.00887	0.188	No. 3A 19	103.1	102.9	0.17	20.7	0.00792	0.164
			No. 3A-5[c]	101.2	101.0	0.2	20.4	0.00829	0.169	No. 3A-20	108.0	102.5	5.4	23.3	0.038	0.885
			No. 3A-6c	124.0	106.5	16.4	38.8	0.0264	1.025	No. 3A-21	123.7	106.2	16.5	30.0	0.0585	1.755
			No. 3A-7	122.1	101.0	20.9	41.3	0.0242	1.00	No. 3A-22	121.6	102.6	18.5	30.0	0.0514	1.54
			No. 3A-8	107.6	103.0	4.5	25.3	0.0284	0.718	No. 3A-23	123.5	102.5	20.5	31.0	0.0520	1.61
			No. 3A-9	87.6	83.5	4.9	20.8	0.0225	0.469	No. 3A-24	107.3	105.0	2.2	22.2	0.0207	0.460
			No. 3A-10[c]	86.4	84.5	2.3	18.8	0.0178	0.335	No. 3A-25	110.5	106.0	4.2	23.4	0.0390	0.912
			No. 3A-11[c]	82.8	91.1	1.9	19.9	0.0177	0.352	No. 3A-26[g]	112.5	111.8	0.66	22.4	0.0118	0.265
			No. 3A-12	111.4	109.0	2.2	24.3	0.0240	0.582	No. 3A-27[g]	112.2	111.1	0.98	22.8	0.0138	0.314
			No. 3A-13	105.6	103.0	2.0	22.7	0.0210	0.476							
			No. 3A-15	91.2	89.3	2.1	19.7	0.0234	0.463							
			No. 3A-16	110.4	105.0	5.1	26.4	0.0294	0.777							
			No. 3A-17	92.7	90.8	2.1	20.1	0.0218	0.437							
Bangor sand and gravel (Well graded, 1½-in. maximum)	2.70	0.20	No. 3B-1	131.3	127.0	3.4	29.8	0.0299	0.890	No. 3B-4[g]	133.6	130.8	2.1	27.5	0.0264	0.725
			No. 3B-2	133.0	131.5	1.1	27.7	0.0243	0.673	No. 3B-5	131.7	127.1	3.6	27.7	0.0411	1.04
			No. 3B-3	138.8	127.0	9.3	36.3	0.0310	1.13	No. 3B-6	143.8	130.8	9.9	32.8	0 0466	1.53
			No. 3B 11	133.7	133.3	0.3	27.1	0.0174	0.472	No 3B-7	140.6	127.1	10.6	32.2	0.0460	1.48
										No. 3B-8	132.5	130.2	1.8	27.2	0.0244	0.665
										No. 3B-9	143.6	130.2	10.3	32.8	0.0450	1.48
										No. 3B-10	133.2	132.9	0.23	26.7	0.0174	0.465
Somerville cinders (Well graded 1-in. maximum)	2.27	0.18	No. 3C-1	73.5	60.9	20.7[e]	23.6	0.0150	0.353	No. 3C-5	67.9	60.8	11.7	14.5	0.0256	0.372
			No. 3C-2	82.0	60.0	36.6	32.8	0.0141	0.462	No. 3C-6	82.4	60.8	35.5	21.7	0.0322	0.700
			No. 3C-3	73.7	60.8	21.2[f]	23.9	0.0148	0.354	No. 3C-7	63.5	63.4	0.09	11.4	0.0133	0.152
			No. 3C-4	68.7	61.7	11.3	18.1	0.0164	0.297							
			No. 3C-8	62.6	61.9	1.1	11.9	0.0145	0.173							
Mystic slag (1½-in. maximum)	2.45	0.17	No. 3D-1	86.3	79.1	9.1	17.5	0.0107	0.188	No. 3D-3	92.0	87.2	5.5	17.2	0.0143	0.245
			No. 3D-2[d]	108.4	81.2	33.5	40.9	0.0135	0.553	No. 3D-4	111.4	87.2	27.7	26.9	0.0250	0.673
			No. 3D-6	92.9	92.3	0.6	16.3	0.00885	0.146	No. 3D-5	89.5	89.3	0.21	15.3	0.00798	0.122

Winchester crushed trap rock ($\frac{3}{4}$-in. maximum)	2.91	0.20	No. 3E-1	101.1	99.2	1.9	21.7	0.0161	0.350	No. 3E-9	104.3	102.8	1.5	21.3	0.0154	0.328
			No. 3E-2	102.1	100.0	2.1	22.1	0.0168	0.371	No. 3E-10[h]	129.3	102.8	25.8	33.8	0.0349	1.18
			No. 3E-3	102.8	98.5	4.4	23.6	0.0171	0.403	No. 3E-11	108.8	106.5	2.2	22.5	0.0185	0.417
			No. 3E-4	125.7	98.5	27.2	46.5	0.0182	0.849	No. 3E-12	104.8	103.6	1.2	21.3	0.0157	0.334
			No. 3E-5[d]	127.5	99.3	28.4	48.0	0.0484	2.32	No. 3E-13[i]	130.0	106.5	22.1	33.1	0.0293	0.989
			No. 3E-6a[d]	127.7	100.0	27.7	47.7	0.0488	1.85	No. 3E-14[h]	129.4	103.5	25.0	33.6	0.0316	1.06
			No. 3E-7	104.6	102.0	2.5	23.0	0.0161	0.371	No. 3E-15	106.8	104.7	2.0	22.0	0.0170	0.375
			No. 3E-8	129.2	102.0	26.7	47.7	0.0310	1.48	No. 3E-17	102.7	102.5	0.21	20.6	0.0763	0.157
			No. 3E-21	112.6	112.4	0.2	22.7	0.00864	0.196	No. 3E-18	111.4	111.3	0.12	22.3	0.0879	0.196
Asphaltic bituminous concrete	2.60	0.20	No. 3F-1[h]	150.0	150.0	0.0	30.3	0.0229	0.691							
			No. 3F-2[i]	150.0	150.0	0.0	30.3	0.0227	0.686							
Blended bituminous concrete aggregates	2.81	0.20	No. 3G-1[c]	133.5	133.5	0.0	26.7	0.0117	0.313							

[a] Assumed.

Minimum unit dry weight, 92.9 lb. per cubic foot; maximum unit dry weight, 110.9 lb. per cubic foot.

[c] Sample not properly sealed, some water leaked into sample during test.

[d] Test results are not consistent with results of other tests and time curves are erratic.

[e] Average water content at top of sample, 14.2; bottom, 27.3.

[f] Nonuniform water content.

[g] Slight leaking into cylinder during test.

[h] Cover lifted off cylinder due to heaving during test. Results slightly affected by leaking of brine into specimen.

[i] Average heave of 0.8 in. measured. Slight leaking.

TEST PROCEDURE

Tests were performed on the materials in both the frozen and unfrozen condition. The samples were of cylindrical shape, 5.36 in. in diameter and 10.68 in. in height, and were contained in a brass cylinder of $\frac{1}{16}$-in. wall thickness. Brass was used because of its high thermal conductivity in comparison with soil. A thermocouple was placed during compaction of a sample at the exact midpoint of the cylinder by use of a template. The material to be tested was placed in approximately five equal layers and compacted, using an increasing number of blows on each layer to obtain a uniform unit weight throughout the sample. Care was taken not to injure nor displace the thermocouple during compaction. After the cylinder was full, the ends were covered with $\frac{1}{16}$-in. brass covers and sealed to prevent leakage during the test. In the unfrozen tests the specimens were immersed in a water bath and brought to a constant temperature of approximately 75 F. The specimens were removed from this bath and immediately immersed in a second water bath at a temperature of approximately 40 F. In the frozen tests the specimens were subjected to a constant freezing temperature of approximately −4 F. inside the freezing cabinet until temperature equilibrium was reached and then immersed in a brine bath at a temperature of approximately 27 F. The resulting temperature changes were measured at the midpoint of the specimens until temperature equilibrium was reached.

BASIS FOR ANALYSIS OF TEST RESULTS

Rigorous solutions for the flow of heat in a cylinder of infinite length and in one of finite length are presented by H. S. Carslaw (8). These equations have been solved for temperature change at the center of a cylinder due to a suddenly impressed temperature change at the surface of the cylinder. These solutions neglect resistance to heat flow at the boundary. The solutions are expressed for convenience in the form of Eq. *a* and two curves, all shown in Fig. 4. Curve 1 is for a cylinder whose height is twice its diameter; curve 2 is for a cylinder of infinite length. These two curves relate the percentage temperature change,[3] and the dimensionless time factor, T; thus, using either curve, values of time factor may be determined for desired values of percentage temperature change.

Since all tests were performed using a cylindrical specimen whose height was twice its diameter, curve 1 was used for analysis. If the ends of a cylinder of any finite length are capped with a material whose thermal conductivity is very small in comparison with the material being tested, then curve 2 may be used. For each test a plot of percentage temperature change *versus* time, Fig. 5, was made. From this plot and curve 1, Fig. 4, the value for thermal diffusivity was computed for 50 per cent temperature change using Eq. *a*, Fig. 4. The 50 per cent value was chosen after a few trial computations indicated that thermal diffusivity varied only slightly with the percentage temperature change. The few tests, for which the time curves indicate a variation of thermal diffusivity with percentage temperature change, were considered unreliable and are not included in the final results.

It can be shown that the volumetric heat capacity of a given unit volume of material is equal to the sum of the volumetric heat capacities of the several component parts. Thus, the volumetric heat capacity of a unit volume of soil equals the sum of the volumetric heats of the dry soil and of water or ice, depending upon its temperature. The volumetric heat capacity of each test specimen was computed from its water content and unit weight, using an assumed value for the specific heat of the dry soil and average values of 1.0 and 0.5 for the specific heat of water and ice, respectively.

TABLE II.—VALUES FOR SPECIFIC HEAT OF SOILS, ROCKS AND MINERALS.

From Table 280, Smithsonian Physical Tables, Eighth Revised Edition, 1933

Substance	Temperature, deg. Cent.	Specific Heat
Asbestos	20 to 98	0.195
Basalt, fine, black	12 to 100	0.1996
Beryl	15 to 99	0.1979
Calcite, $CaCO_3$	0 to 50	0.1877
Chalcopyrite	15 to 99	0.1291
Cryolite	16 to 99	0.2522
Dolomite	20 to 98	0.222
Fluorite, CaF_2	15 to 99	0.2154
Garnet	16 to 100	0.1758
Gneiss	17 to 99	0 196
Granite	12 to 100	0.192
Hematite, Fe_2O_3	15 to 99	0.1645
Hornblende	20 to 98	0.1952
Hypersthene	20 to 98	0.1914
Kaolin	20 to 98	0.224
Labradorite	20 to 98	0.1949
Lava, Aetna	23 to 100	0.201
Lava, Kilauea	25 to 100	0.197
Limestone	15 to 100	0.216
Magnetite	18 to 45	0.156
Marble	0 to 100	0.21
Mica (Mg)	20 to 98	0.2061
Orthoclase	15 to 99	0.1877
Quartz	12 to 100	0.188
Quartz	0	0.1737
Quartz sand	20 to 98	0.191
Rock-salt	13 to 45	0.219
Sandstone	....	0.22
Serpentine	16 to 98	0.2586
Siderite	9 to 98	0.1934
Talc	20 to 98	0.2092

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[3] *List of Definitions:*

Term	Symbol	Definition
Unit weight		Weight per unit of volume of material as tested.
Unit dry weight	γ	Weight per unit of volume of solids.
Specific heat	c_1	Ratio of the quantity of heat required to raise unit mass of the substance 1 C. to the quantity required to raise unit mass of water 1 C.
Thermal conductivity	k	The quantity of heat that will pass through a unit area of unit thickness in unit time under a unit temperature gradient.
Thermal diffusivity	a	The ratio of the thermal conductivity to the volumetric heat capacity.
Volumetric heat capacity	C	The heat in Btu. required to raise 1 cu. ft. of soil 1 F.
Percentage temperature change	u	Ratio, expressed as a percentage, of the difference between the initial uniform temperature and the temperature at the center of the cylinder at a given time to the difference between the initial uniform temperature and the suddenly impressed exterior temperature.

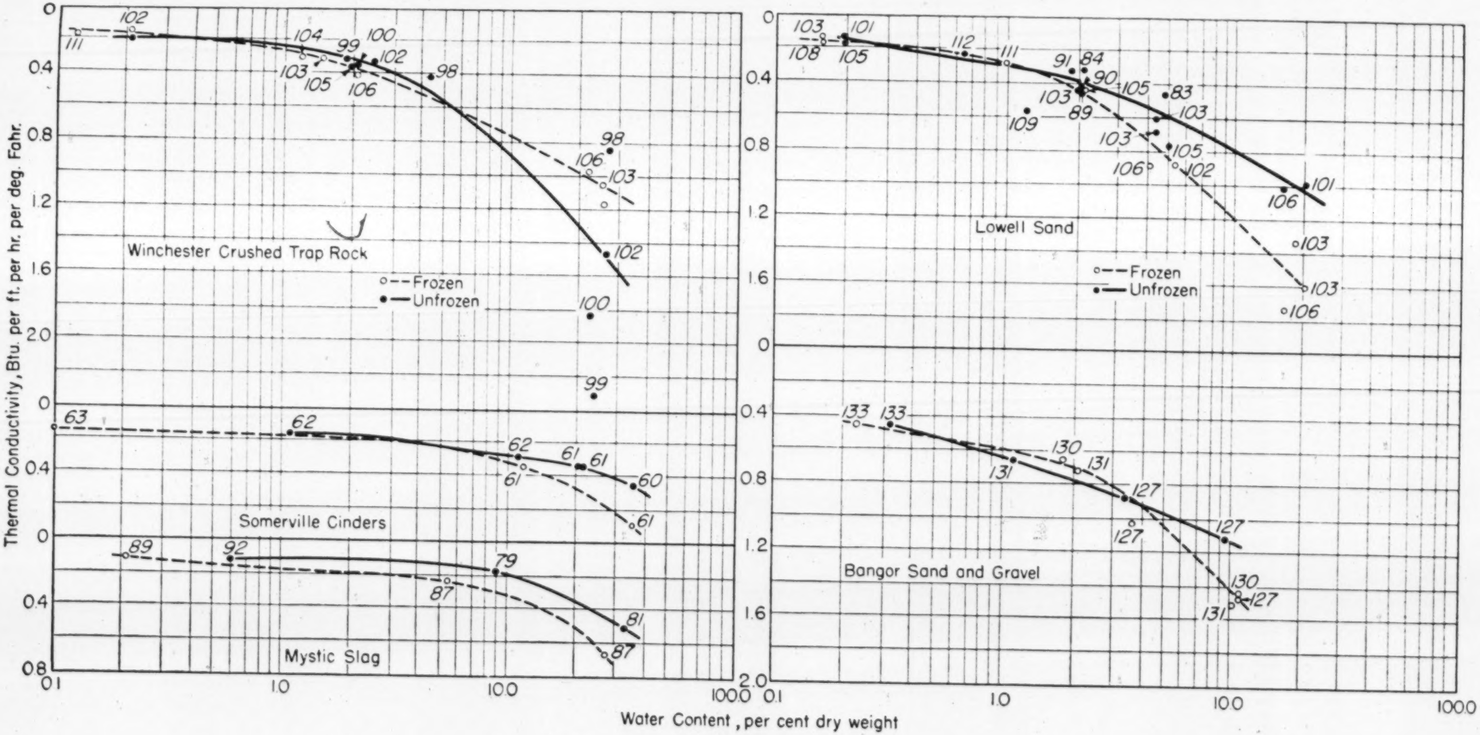


Fig. 6.—Thermal Conductivity *versus* Water Content.

Equations *b* and *c*, Fig. 4, were used for computing values of the volumetric heat capacity.

Tests by the Bureau of Reclamation(7) for the specific heat of six different rocks and concrete from eight different locations indicate that the specific heat of the samples tested changed approximately 20 per cent with a 100 F. change in temperature, and the average values of the specific heat for all samples tested was approximately 0.22 for concrete and 0.24 for rocks.

There are tabulated in Table II selected values for the specific heat of various minerals, soils, and rocks, as listed in Smithsonian Physical Tables, Eighth Revised Edition, 1933.

These data, together with the tests performed by the Bureau of Reclamation, indicate that the specific heat of most soils and rocks is approximately a constant. The values for specific heat shown in Table I were assumed based upon these data.

From the tested values for the thermal diffusivity, values for the thermal conductivity were determined, utilizing the computed values for the volumetric heat capacity.

RESULTS AND CONCLUSIONS

The results of the tests conducted are summarized in Table I and Fig. 6. For the purpose of comparison of results, values of thermal conductivity are used in preference to values of thermal diffusivity, since the thermal diffusivity is not often a convenient term for use in studies of temperature conditions within a soil mass.

There was some doubt regarding the accuracy of the values determined for thermal conductivity using the method described, hence arrangements were made for a series of tests on the Lowell sand by the U. S. Engineer Office, St. Paul, Minn. These tests were performed, in connection with their investigations of permafrost, at the University of Minnesota. The method used consisted essentially of measuring the quantity of heat conducted through a sample of soil exposed to a constant uniform temperature gradient. Tests were performed at various water contents and unit weights. A comparison of the values of thermal conductivity obtained by the two methods, contained in Table III, indicates a satisfactory agreement.

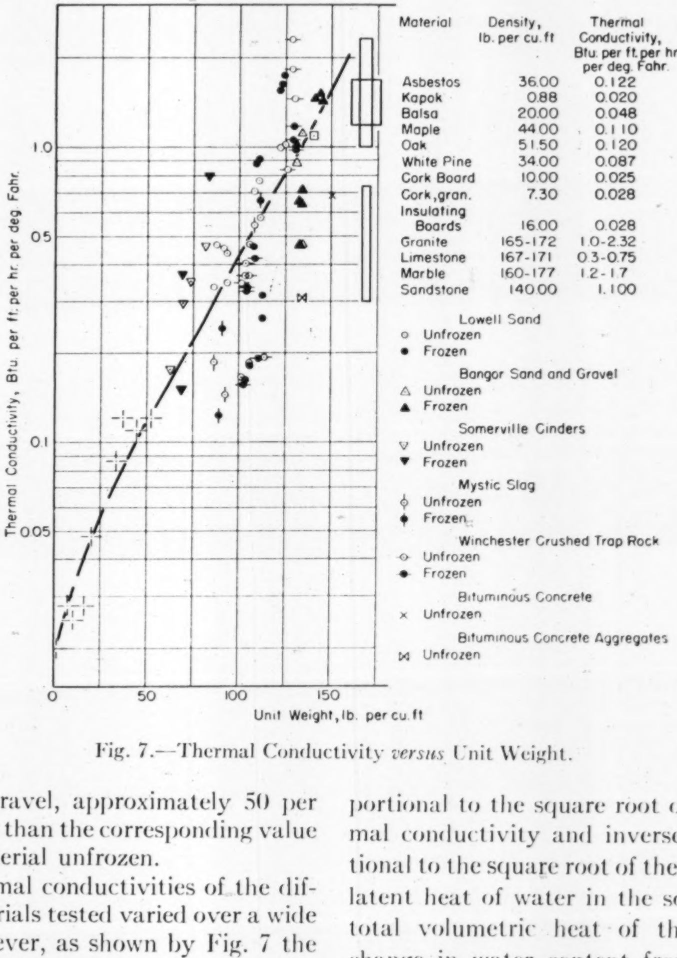
TABLE III.—COMPARISON OF VALUES OF THERMAL CONDUCTIVITY DETERMINED BY TWO DIFFERENT METHODS.

Laboratory and Test Method	Average Water Content, per cent	Average Unit Dry Weight, lb. per cu. ft.	Condition of Test	Average Thermal Conductivity, Btu. per ft. per hr. per deg. Fahr.
Boston[a]	0.2	101	Unfrozen	0.15
Minnesota	0.2	100	Unfrozen	0.16
Boston	0.2	105	Unfrozen	0.18
Minnesota	0.2	105	Unfrozen	0.19
Boston	0.16	104	Frozen	0.16
Minnesota	0.2	102	Frozen	0.17
Boston	4.8	104	Unfrozen	0.69
Minnesota	4.4	106	Unfrozen	0.86
Boston	4.8	104	Frozen	0.90
Minnesota	4.4	106	Frozen	0.68
Boston	11.0	103	Frozen	1.17
Minnesota	10.8	105	Frozen	1.15

[a] Method used consisted of applying a sudden external temperature change to a cylindrical specimen and measuring the resulting temperature change at center of cylinder as a function of time.

[b] Method used consisted of applying a constant temperature gradient to a hollow cylindrical test specimen and measuring the resulting quantity of heat conducted as a function of time.

As shown by Fig. 6, the thermal conductivity of the soils tested depends primarily upon the water content and its condition, that is, whether frozen or unfrozen. As might be expected, the thermal conductivity of the material frozen and unfrozen approaches a common value as the water content approaches zero. With increasing water content the thermal conductivity increases in magnitude. For low water contents, from about 1 to 5 per cent, the thermal conductivity of the frozen soil is slightly less for some of the materials tested than the value for the material unfrozen at the same water content. For high water contents, the thermal conductivity of the frozen material is, for all except Bangor sand and gravel, approximately 50 per cent greater than the corresponding value for the material unfrozen.



Material	Density, lb. per cu. ft	Thermal Conductivity, Btu. per ft. per hr. per deg. Fahr.
Asbestos	36.00	0.122
Kapok	0.88	0.020
Balsa	20.00	0.048
Maple	44.00	0.110
Oak	51.50	0.120
White Pine	34.00	0.087
Cork Board	10.00	0.025
Cork, gran.	7.30	0.028
Insulating Boards	16.00	0.028
Granite	165-172	1.0-2.32
Limestone	167-171	0.3-0.75
Marble	160-177	1.2-1.7
Sandstone	140.00	1.100

Fig. 7.—Thermal Conductivity *versus* Unit Weight.

The thermal conductivities of the different materials tested varied over a wide range; however, as shown by Fig. 7 the thermal conductivity is approximately related to unit weight for all materials tested. In addition, on Fig. 7 are plotted values for the thermal conductivity of selected rocks and insulators.

From the standpoint of thermal conductivity, it might be concluded that the frost penetration into a dry soil would be less than that into the same soil in a saturated condition. Unfortunately this is not the case, for it can be shown that the depth of frost penetration, other things being equal, is approximately proportional to the square root of the thermal conductivity and inversely proportional to the square root of the sum of the latent heat of water in the soil and the total volumetric heat of the soil. A change in water content from 5 to 10 per cent will increase the sum of the volumetric heat and latent heat by about 100 per cent, while the same change in water content will increase the thermal conductivity only about 20 per cent. Thus the depth of frost penetration will generally be greatest in a given soil at zero water content and will become less with increasing water content. Berggren (6) arrived at this same general conclusion.

Acknowledgment:

The investigation reported herein was performed by the Office of the Division Engineer, New England Division, Corps of Engineers, for the Office, Chief of Engineers, Washington, D. C. The tests were performed in the Soil Mechanics Cold Room of the Graduate School of Engineering, Harvard University, the use of which was made possible through the courtesy of Professor Arthur Casagrande. The solutions of the equations for the flow of heat in a cylinder, together with the computations for the values of time factor for selected values of percentage temperature change, were made by Reginald A. Barron. The test program at the University of Minnesota was under the supervision of Major H. J. Manger of the Office of the District Engineer, St. Paul, Minn., Corps of Engineers. The tests at the University of Minnesota were under the general direction of Professors F. B. Rowley, A. B. Algren, and M. S. Kersten.

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DISCUSSION

MR. J. O. OSTERBERG.[1]—I should like to ask a question. Are the values given for thermal diffusivity in relation to density for zero water content?

MR. WILLIAM L. SHANNON (*author*).—The values given for thermal conductivity are for the unit weights as tested.

MR. OSTERBERG.—How can a generalized picture be given when the water content can vary so considerably? I thought that the values given were the values of constant thermal diffusivity as the water content approaches zero.

MR. SHANNON.—The results, as contained in Fig. 7, are presented only for the purpose of indicating the general trend. This figure indicates that unit weight is of secondary influence to water content for any given material.

MR. OSTERBERG.—I wonder whether it would be possible to give values of thermal conductivity for zero water content for different dry-densities and then using a shaped curve, such as you have, construct approximately what it would be for various water contents.

MR. SHANNON.—From Fig. 7, it can be seen that each group of data falls in a band which has a slope considerably steeper than the generalized curve. In any group the water content varies from a low value for tests having low thermal conductivity to a high value for tests having a large thermal conductivity. From the data presented, the curves which you suggest could probably be constructed. Perhaps use of degree of saturation in place of water content would result in a better correlation. It would appear desirable, in formulating such a relation, to include the detailed data on thermal conductivity now being obtained by the University of Minnesota.

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[1] Assistant Professor of Civil Engineering, Technological Institute, Northwestern University, Evanston, Ill.

UPLIFT SOIL PRESSURE ON BRIDGE FOUNDATIONS AS REVEALED BY SHEAR TESTS*

By G. O. Kerkhoff[1] and W. S. Housel[1]

This paper presents the measured uplift soil pressures developed in soft plastic blue clay at a large railroad-expressway grade separation in Detroit. In 1944, during construction, six pressure cells were placed under the heavily reinforced concrete slab that strutted the abutment and pier footings. The design of this structure was based on the results of "ring shear" tests made by the Michigan State Highway Department in the laboratory at Ann Arbor. Uplift pressures were based on the yield value shearing resistance, and a substantial overload ratio was used on the assumption that some progressive movement of the clay would be permissible. Even then the reinforced concrete slab designed to resist uplift appeared so heavy, judged by usual standards, that the designing engineers were reluctant to accept the predicted uplift pressures without further demonstration.

During the early stages of construction, cell readings registered surcharge of the earth embankment at the abutment and also surcharge of the fill in the future south lane span. Within the first six months uplift pressures increased to a maximum of 2400 to 2700 lb. per sq. ft. and maintained this high initial pressure for about one year and then gradually began to decrease to an average uplift pressure of about 2000 lb. per sq. ft. up to 30 months. Within the first four months the heavily reinforced strut slabs between the abutments and the north and south pier developed identical pattern of cracking—surface cracks opened up about $\frac{1}{4}$ in. the full width and length of each slab.

Analysis of enbankment stability of the abutments shows an excellent correlation between computed and measured cell pressures. Based on the yield value shearing resistance the maximum predicted uplift pressure was 2465 lb. per sq. ft. ignoring any lateral distribution of the vertical load of the abutment fill. Assuming the maximum amount of lateral distribution of this vertical pressure, the minimum predicted uplift pressure was 1758 lb. per sq. ft. Thus the comparison of computed and measured uplift pressure demonstrates, first, the relative accuracy of laboratory shear tests in plastic clay as a basis of design and, second, that static equilibrium in the clay mass requires that shearing stresses be held down to the yield value.

This paper also presents the results of two similar projects which were conducted at the same time. One is a railroad-expressway grade separation of two spans with low fills at the abutments. The other is a small, box-like grade separation of one span with low fills at the abutments. The results obtained indicate that measured pressures are substantially less than those computed on the basis of the yield value from shear tests. It is thought that this discrepancy may be due to the effect of indeterminate factors in the stability such as the confining influence of pile groups that support the footings or possible continued movement of the soil mass. Additional studies are being made of these factors.

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* Presented at the Fiftieth Annual Meeting of the Scoiety, June 16–20, 1947.

[1] Soil Testing Engineer and Research Consultant, respectively, Michigan State Highway Department; and Assistant Professor of Civil Engineering and Associate Professor of Civil Engineering, respectively, University of Michigan, Ann Arbor, Michigan.

EMBANKMENT STABILITY IN PLASTIC CLAY SOIL

The stability of abutment fills on plastic clay soil at grade separation structures and stability of embankment slopes in such clay became a major design problem in the River Rouge section of the Detroit Industrial Expressway. In addition to the foundation support for the structure's footings, consideration of horizontal thrust at the abutments and uplift between abutment and pier footings was found necessary. The solution to these two problems was to construct the reinforced concrete slab between the abutments and pier footings. The purpose of the slab is to act as a strut between footings to take horizontal thrust and also to resist uplift soil pressures. The thickness of a slab required to resist the computed uplift soil pressures at the larger railroad-expressway grade separations seemed out of line with empirical practice in this area. After some study it was decided to construct a 24-in. heavy, reinforced concrete slab and tie it in to the abutment and pier footings. In order to check this design a research project was set up to measure the actual uplift pressures. Such information was desired as an aid in the design of extension of the expressway system in the City of Detroit.

The stability of embankment slopes in the depressed roadway section decreases rapidly with increased depth of excavation. This was an important control in establishing expressway grade and the required width of right of way. Because of the many factors involved it was sometimes necessary to design embankment slopes in which the computed stress in the embankment is greater than the shearing resistance (yield value) of the cohesive soil. However, this index or "overload ratio" is kept as close to unity as the circumstances permit and definite limits, which are dangerous to exceed, have been tentatively established.

GENERAL SOIL CONDITIONS

This area, geologically, is part of a dry lake bed, typical of soft glacial-lake deposits bordering the Great Lakes. The subsoil is a deep lacustrine deposit of cohesive clay. At the surface the subsoil has developed into a firm yellow clay to a maximum depth of about ten feet. Below the firm surface layer the soil is a soft plastic blue clay to a few feet above bedrock. The physical properties and structure of the subsoil from the surface to a depth of about 40 ft. are quite variable. Below a depth of 40 ft. the soil is often very uniform in every respect. Bed rock averages about 110 ft. below the surface.

PRESSURE-CELL INSTALLATION

Three grade separations were selected for the proposed research project, the New York Central Railroad main line crossing, the Pere Marquette Railroad main line crossing, and the Michigan Avenue Interchange. During construction in the summer of 1944, a total of thirteen Goldbeck pressure cells were installed below the subbase slabs. Although other types of pressure cells were investigated, only the Goldbeck type of cell was available at that time. Three cells were placed between the center pier and east abutment at the

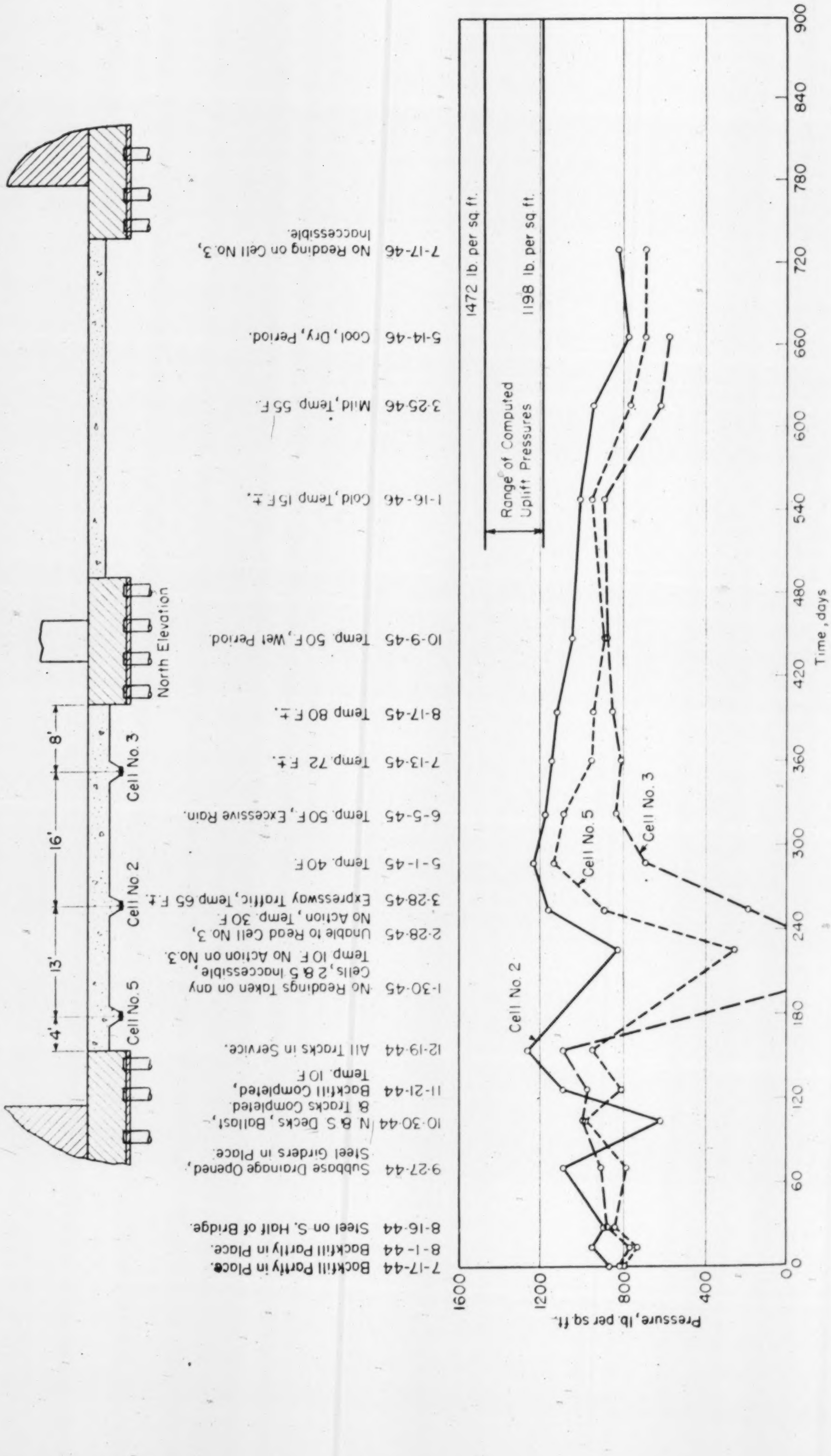

FIG. 1.—Pressure Cell Readings at the N.Y.C.R.R.-D.I.E. Structure.
Goldbeck Soil Pressure Cell Readings.

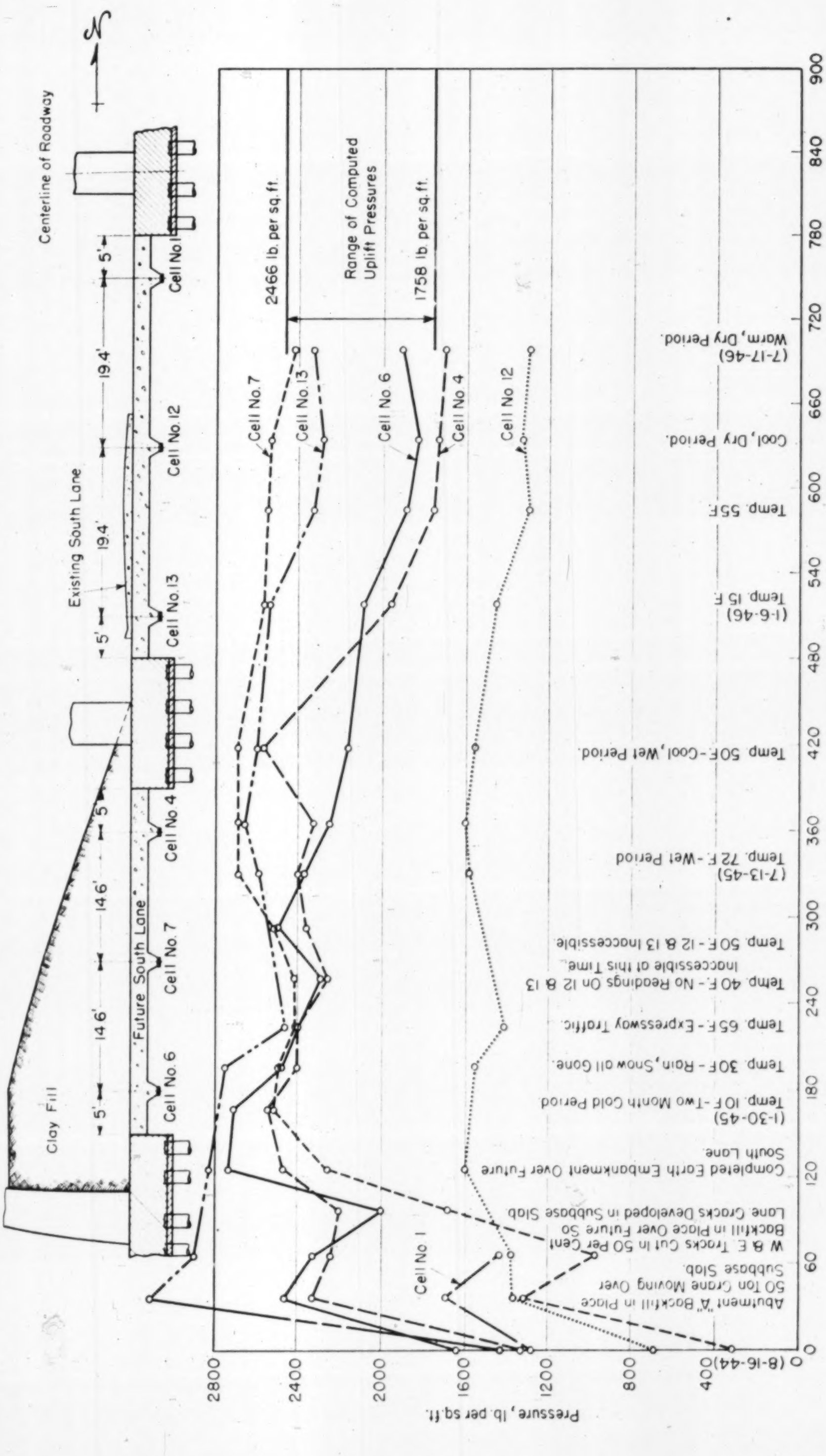

FIG 2 —Pressure Cell Readings at the P.M.R.R.-D.I.E. Structure.

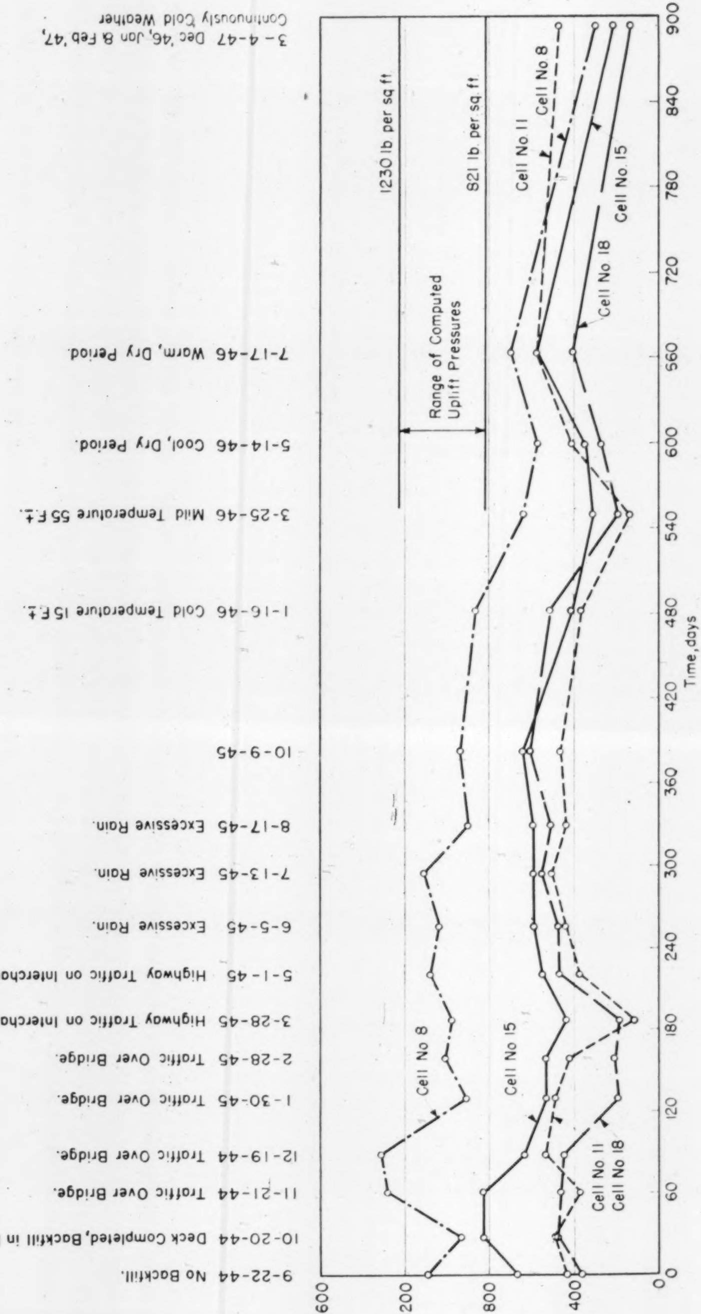

FIG. 3.—Pressure Cell Readings at the Michigan Avenue-D.I.E., Interchange.

N.Y.C.R.R. crossing; six cells were placed at the P.M.R.R. crossing, three between the center pier and the south pier and three between the south pier and the south abutment; and four cells were placed at the Michigan Avenue Interchange, two at the center of the structure on centerline of Michigan Avenue and two near the south end of the structure. At each structure the cells were well seated in the soft blue clay with the weighing face set down in the clay. The back of the cells were covered with concrete at the time of pouring of the slab. Each cell was connected to a reading station by two sections of $\frac{3}{8}$-in. diameter soft copper tubing. Only one tube connection was actually necessary but the two tube connections to each cell permit the circulation of air in the system to remove any condensation. At the New York Central and Pere Marquette structures the cells were placed approximately 6 in. below the bottom of the slab. At the Michigan Avenue Interchange cells were placed at different elevations below the slab because of irregular excavating and backfilling.

MEASURED UPLIFT PRESSURES

Readings of the pressure cell were made every month for the first year and were continued thereafter at random time intervals. All cells have functioned satisfactorily, but cell No. 1 was lost at the P.M.R.R. structure when its reading station was destroyed. Charts of pressure cell readings are shown in Figs. 1, 2, and 3.

As shown by Fig. 1, the registered cell pressures at the N.Y.C.R.R. structure start at approximately 800 lb. per sq. ft. and maintain this and slightly higher pressure for eighteen months. After this period the cells show a small decrease in pressure, leveling off to an average of about 700 lb. per sq. ft. after 2 yr. It is interesting to note that during the first winter the cells registered a marked decrease in pressure, but regained their former pressure with the advent of warmer temperatures. This rather unusual behavior is typical of all three installations and is definitely associated with low temperatures and the period of maximum frost heaving in late winter. Frost penetration of over 3 ft. would be required to freeze the soil below the cell, so it is considered doubtful that the soil around the cells was actually frozen.

The pressure cell readings at the P.M.R.R. structure, shown in Fig. 2 were begun one day after their installation. All initial readings were very much lower than the following readings for the first year. This set of soil pressure curves shows that completion of the fill at the abutments is reflected in a sharp increase in uplift pressure below the strutted slabs. Apparently during the following period adjustments were taking place resulting in a decrease in cell pressure. During this readjustment stage the subbase slab developed a visible pattern of cracking. For this same period, up to 120 days, cell No. 7 at the center of the slab span did not show as high a pressure intensity as cells Nos. 6 and 4. This is probably due to upward flexing of the slab. When this slab was confined by a clay fill after some 120 days the pressure at cell No. 7 increased to a maximum. After this change lower pressure on cells Nos. 6 and 4 may reflect the confining influence of pile groups supporting the footings.

The initial cell pressures in the future south lane slab were 1600 lb. per sq. ft., but after completion of the fill back of the abutment the cells registered a pressure of 2400 lb. per sq. ft. It is believed that the slab failed at this uplift pressure, accounting for the sharp decrease in pressure at about 60 days on all cells except Nos. 12 and 13. The cracking pattern consisted of two $\frac{1}{4}$-in. openings at the surface, one across the full width of the slab between cell No. 7 and No. 4 and about parallel to the face of the abutment, and the other crack about perpendicular to the face of the abutment at the west two-thirds of its width. The pattern of cracking was identical at the future north lane slab adjacent to the north abutment. At about 240 days the four cells nearest to the abutment show about the same pressures coincident with a low point on all graphs characteristic of a late winter reading. After this there was a slow gradual decrease in pressure. Cells Nos. 7 and 13 follow a similar pressure pattern and cells Nos. 4 and 6 follow a similar pattern but at a lower pressure intensity. At the end of two years the maximum pressure is 2400 lb. per sq. ft. and the minimum is 1700 lb. per sq. ft. Cell No. 12 in the center of the slab span in the present south lane registered a much lower pressure intensity, its maximum pressure being 1600 lb. per sq. ft. and its pressure at two years being 1300 lb. per sq. ft.

The pressure cell readings at the Michigan Avenue Interchange as shown in Fig. 3 are of a much lower intensity. The registered soil pressures are more scattered but show a somewhat uniform pressure at 660 days. Cell No. 8 located in the center of the slab span and on the centerline of Michigan Avenue shows a much higher pressure than the other three cells. The pressure curves show the same general pattern of a slow gradual decrease in uplift pressure which after some two years may be leveling off. The temporary decreases in cell pressures during the two winter periods are again evident. At two years the uplift pressures average about 500 lb. per sq. ft.

SHEARING RESISTANCE TESTS

The use of shear tests has become routine procedure in the Michigan State Highway Department and shear values are considered required design information by both the Bridge Design and Road Design Divisions. Field sampling and laboratory testing have also developed into routine procedure and have been set up on a production basis. The volume of such testing has now reached the level of several hundred tests per month. The so-called "ring shear test" developed at the University of Michigan and described in detail in the A.S.T.M. Proceedings for 1939[2] has been used without any significant change for the past 15 years other than for some refinement in equipment.

Under present practice the core barrel for taking the $1\frac{3}{8}$-in. core sample is operated in a 2-in. diameter casing which is driven or jetted down to the depth of sampling. The core barrel is driven or pushed into undisturbed soil below the bottom of the casing where the sample is taken. Samples are obtained at each significant change in soil strata and otherwise at depth intervals of 5 ft. Samples are shipped immediately to the laboratory in metal containers sealed against loss of moisture. One of the features of sampling and testing that has been emphasized is extreme care in handling of the sample to protect it from any disturbance or exposure. Under present practice the sample is completely sealed against exposure and untouched by the operator from the time it is taken from the ground until the shear test is completed.

For the past five years the University of Michigan laboratory has been conducting an unconfined compression test on a section of the same core sample as a check test. The procedure followed is that used by University of Illinois laboratory and is regarded by the authors as a rapid shear test. The equivalent shearing resistance is taken as one half the unconfined compression value at 20 per cent vertical deformation. The load

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[2] W. S. Housel, "The Shearing of Soil—Its Measurement and Practical Significance," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 1084 (1939).

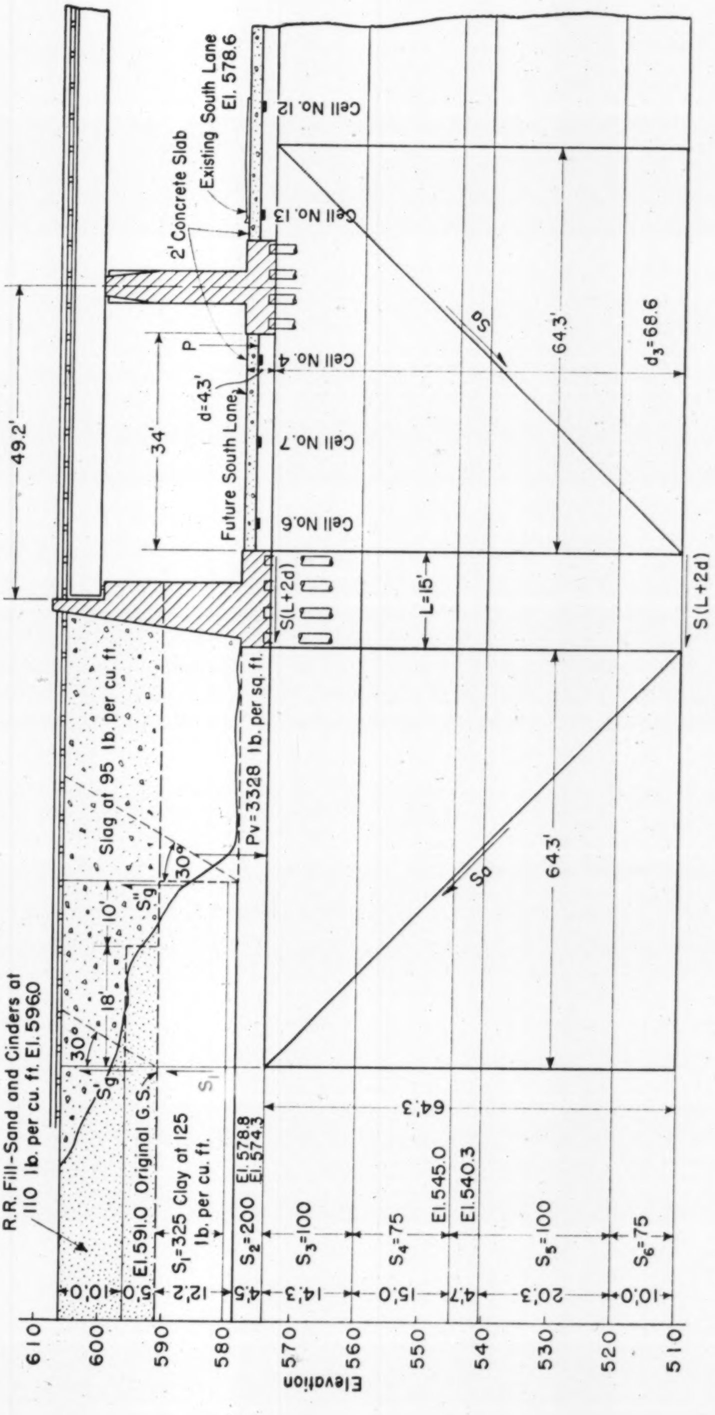
R.R. Fill-Sand and Cinders at 110 lb. per cu. ft. El. 596.0
El. 591.0 Original G.S.
S1=325 Clay at 125 lb. per cu. ft.
S2=200
El. 578.8
El. 574.3
S3=100
S4=75
El. 545.0
El. 540.3
S5=100
S6=75
10.0
5.0
12.2
4.5
14.3
15.0
4.7
20.3
10.0
610
600
590
580
570
560
550
540
530
520
510
Elevation
64.3'
S'g
S''g
S1
30°
18'
10'
30°
Slag at 95 lb. per cu. ft.
Pv=3328 lb. per sq. ft.
So
64.3'
S(L+2d)
L=15'
S(L+2d)
49.2'
34'
Future South Lane
d=4.3'
P
2' Concrete Slab
Existing South Lane El. 578.6
Cell No. 6
Cell No. 7
Cell No. 4
Cell No. 13
Cell No. 12
So
64.3'
d3=68.6

FIG. 4.—Diagram Showing Stability Controlled by Depth to Hardpan.

increments are applied at ½-min. intervals and are selected to cause complete failure in 5 min. Shear values from the unconfined compression test in plastic clays are approximately four times the yield value from the ring shear test. However, this ratio is erratic and varies through a wide range for stiff clays and those having some granular characteristics.

On the usual construction project several or perhaps a large number of borings will be required depending on the size of the project. The next step is to prepare a composite shear profile of all comparable borings and select recommended shear values for design. The recommended shear values are determined by visual averaging of results to give values representative of the various strata in the group of borings.

COMPUTED UPLIFT PRESSURES

The data obtained at the P.M.R.R.-D.I.E. structure have been used to make a typical analysis of the three grade separations studied. Typical computation for uplift pressures is included in the Appendix[2a] and illustrated in Fig. 4.

In Part I the computations are made for an element of mass the size of which is determined by the greatest depth of soft clay, or depth to hardpan as shown in Fig. 4. This size of element also embraces the four cells nearest to the abutment, which have shown similar pressure intensities and behavior as illustrated in Fig. 2. The uplift pressure at the bottom of the slab for this large element is 2466 lb. per sq. ft. This uplift pressure is almost identical with the 8-month registered pressure of cells 6, 7, 4, and 13.

It was noted in the discussion of pressure cell readings that immediately after placing of the fill at the abutment, cells registered their highest pressures, but subsequent readings showed a uniform decrease in pressure. It is estimated that during this adjustment period some of the surcharge of the abutment fill is transmitted to the footings supported by piles and to the soil mass by means of shear at planes outlined by the edge of footings within the abutment.

In Part II is shown the computed decrease in effective weight of the abutment fill for a 64.3-ft. element. This decrease is considered due to shearing resistance developed along the perimeter of the footings and in a soil mass through a section 64 ft. back of the abutment and amounts to 708 lb. per sq. ft.

Therefore, the minimum computed uplift pressure at the bottom of the slab is 1758 lb. per sq. ft. This value compares well with the two lowest registered cell pressures of the four cells within the sphere of the large element analysis.

On the other two projects, the N.Y.C.R.R. main line structure and the Michigan Avenue Interchange, the active uplift pressure on the subbase slabs was computed by two methods. At the N.Y.C.R.R. main line structure the controlling condition appears to be displacement of an element of mass of dimensions equal to the clear opening between the abutment and pier footing. For this case the uplift pressure ranges from 1198 to 1472 lb. per sq. ft. depending upon the amount of lateral distribution of the weight of the abutment fill. Comparing these values with the registered cell pressures shown in Fig. 1 it is apparent that only cell No. 2 approached the minimum computed uplift pressures. Cell No. 2 registered its highest pressures during the first eleven months. The reason for the difference between the measured and computed uplift pressures has not been definitely determined. There are certain other factors contributing to the stability of the embankment which are not subject to analysis by present data. It is possible that the large pile group under the abutment and center pier footings are contributing to the stability, particularly on the smaller structure involved in this case. It is also possible that the continued progressive movement of the soil mass maintains a shearing stress greater than the yield value used in the computations. This possibility is being studied but complete data are not presently available.

[2a] See p. 1065.

In the case of the Michigan Avenue Interchange computed uplift pressures ranged from 821 to 1230 lb. per sq. ft. again being controlled by the clear opening between abutment footings. As shown in Fig. 3, only cell No. 8 registered pressure in the computed pressure ranges. The early readings are equal to the maximum computed uplift pressures and up to 16 months leveled off to the computed minimum pressures. At 22 months all cells registered an average pressure of 600 lb. per sq. ft.

Conclusion

The investigation of uplift soil pressures at the Pere Marquette R.R. main line-Detroit Industrial Expressway crossing show an excellent correlation between cell readings and computed pressures. Due to the high abutment fill at this structure the embankment stability proved to be the most critical, thus under this more sensitive condition pressure readings show a definite correlation with the loading of the abutment fill, readjustment stage due to flexing of the slab, and the loading of the fill in the future south lane. The failure of the uplift resistance slabs adjacent to the north and south abutment based on observation of identical pattern of cracking is supporting evidence of the intensity of the uplift pressures.

This investigation demonstrates that uplift soil pressure under bridge foundations can be determined by shearing resistance. It reveals that standard sampling operations and laboratory testing procedure are reliable. Also this investigation demonstrates that the application of shearing resistance to problems in design and construction is practicable. While agreement between computed and measured uplift pressure on the other two projects is not as striking, there is evidence that this is due to other factors not readily susceptible to analysis with present data rather than discrepancy in shear test data or analysis of known factors. The studies of all three projects are being continued to determine changes of behavior, if any, in future years.

The information gained from this research project is considered invaluable and is being used in the design of the extension of the expressway system in the City of Detroit and Wayne County. In the present design of the Edsel Ford and the John C. Lodge expressways the use of shearing resistance in foundation and embankment stability is one of the most important phases of design. At grade separations where uplift pressure requires a resisting slab, it is now designed as a tension member entirely and tied to the footings, thus transmitting uplift pressures to the footings and pile groups.

APPENDIX

Typical Uplift Soil Pressure Computations P.M.R.R. Main Line-D.I.E. Crossing Project X7 of 82-22-10

Part I.—Investigate an element of mass the size of which is determined by the greatest depth of soft clay, or the depth to hardpan. Solve for the unbalanced pressure

P_v' and uplift pressure P_1 at the bottom of the slab (See Fig. 4).

Conditions: The abutment footing will resist horizontal pressure through struts above elevation 574.3.

Elevation 510 (64.3 ft. element)

$$P_v = 2S_a + \frac{S_3(L + 2d_1) + S_6(L + 2d_2)}{64.3} + 2S_a + P_v'$$

where

S_a = average shearing resistance (yield value) of the cohesive soil

$$S_a = \frac{(14.3 \times 100) + (15 \times 75) + (25 \times 100) + (10 \times 75)}{64.3}$$

$= 90$ lb. per sq. ft.

$P_v = \Sigma wh$ (average weight of fill back of abutment)

$$= \frac{(64 \times 10 \times 95)}{64} + \frac{(46 \times 5 \times 95)}{64} + \frac{(36 \times 12.2 \times 95)}{64} + \frac{(18 \times 5 \times 110)}{64} + \frac{(28 \times 12.2 \times 125)}{64} + \frac{(64 \times 4.5 \times 125)}{64}$$

$= 950 + 341 + 652 + 155 + 667 + 563$

$= 3328$ lb. per sq. ft.

$$3328 = (2 \times 90) + \frac{100[15 + (2 \times 4.3)] + 75[15 + (2 \times 68.6)]}{64.3} + (2 \times 90) + P_v'$$

$= 180 + 214 + 180 + P_v'$

$P_v' = 2754$ lb. per sq. ft.

$P_1 = P_v' - wh$

$P_1 = 2754 - 288 = 2466$ lb. per sq. ft. (Maximum uplift pressure)

Part II.—Consider shearing resistance developed by fill within abutment and wing walls along perimeter of footings and in the soil mass through a section 64 ft. back of abutment. Determine amount decrease in unit *wh* pressure for abutment fill, and show decrease in uplift pressure as computed in first part of the computations.

Assume perimeter shear down to elevation 578.8.

Computation of average granular shear values, $S_g' + S_g''$ by shearing resistance from internal friction.[3]

$$S_f = p_h \tan \phi$$
$$p_h = p_v \tan^2 \theta$$
$$S_f = p_v \tan^2 \theta \tan \phi$$
$$S_f = \tfrac{1}{3} w_a h \times 1$$

Assume Theta $\theta = 30$ deg.
Phi $\phi = 45$ deg.

The average p_h pressure can be used as the shearing resistance for the maximum angle of friction of 45 degrees.

$$S_g' = \tfrac{1}{2} \times \tfrac{1}{3} w_a h \times 1$$

$$S_g' = \tfrac{1}{6} w_a h$$

$$= \frac{1}{6} \times \frac{[(95 \times 10) + (110 \times 5)] \times 18}{28} + \frac{1}{6} \times \frac{(95 \times 15) \times 10}{28}$$

$= 160.7 + 84.8$

$= 246$ lb. per sq. ft. (Shear for granular material)

$S_g'' = \tfrac{1}{2} \times \tfrac{1}{3} \times wh = \tfrac{1}{6} \times 95 \times 27.2$

$= 431$ lb. per sq. ft.

$S_l = 325$ lb. per sq. ft. (Shear value for cohesive soil strata)

Perimeter Shear Force

$F = \Sigma$ (Perimeter surface area $\times$ shear)
$= 2{,}594{,}536$ lb.[4]

Unit *wh* pressure decrease due to perimeter shear

$$S_p = \frac{F}{\text{Area}} = \frac{2{,}804{,}746^{4}}{3665}$$

$= 708$ lb. per sq. ft.

Referring to computed uplift pressure P_1 shows decrease due to perimeter shear of the abutment fill.

$P_1 = 2466$ lb. per sq. ft. Decrease by 708 lb. per sq. ft.

$P_2 = 1758$ lb. per sq. ft., Minimum uplift pressure.

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[3] W. S. Housel, "Internal Stability of Granular Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 440 (1936).

[4] Figures are dimensions obtained from plan of abutment footings, not included in this paper.

THE USE OF THE MAXIMUM PRINCIPAL STRESS RATIO AS THE FAILURE CRITERION IN EVALUATING TRIAXIAL SHEAR TESTS ON EARTH MATERIALS*

By W. G. Holtz[1]

Synopsis

Recent research studies by the Bureau of Reclamation on the shear characteristics of soils led to the selection of the maximum principal stress ratio as the failure criterion for evaluating triaxial shear tests. The analysis of triaxial shear-test data showed that the maximum principal stress ratio was the most critical stress condition on a cylindrical specimen subjected to axial load with lateral support. This analysis and the correlation of the maximum principal stress ratio with the minimum volume and maximum pore-pressure condition of triaxial shear specimens are the bases for the acceptance of the failure criterion. This paper also includes a discussion of the testing equipment and procedures used to obtain the test data and a discussion of the effect of pore pressure as related to the analysis of triaxial shear-test results.

The purpose of this paper is to present one of the findings which we obtained as a result of recent triaxial shear and pore-pressure studies on remolded earth materials. This research program, which was performed in the Earth Materials Laboratory of the Bureau of Reclamation, Denver, Colo., from 1944 to 1946, was planned to study the "true" shearing resistance of five types of soils which were molded at several conditions of moisture and density. The values obtained were corrected for pore pressure on the basis of actual pore-pressure measurements taken during the tests. Descriptions of the five soils tested are given in Table I.

Prior to this research program, the maximum deviator stress (the axial stress applied in excess of the lateral stress, $\sigma_1-\sigma_3$) was considered by us to indicate the failure condition of a cylindrical specimen subjected to axial compression with lateral support. It has been a common practice among most investigators to use this failure criterion. However, as we progressed in the research program, it was found that the failure criterion was a critical factor in evaluating research data on triaxial shear tests and that the maximum deviator stress did not represent the true failure condition for all types of soils and testing conditions. The loading of a shear specimen usually produces consolidation which must be accompanied by an extrusion of the pore fluid from the specimen or by the development of pore pressure as the fluid is compressed. When triaxial shear tests are conducted on pervious soils where complete drainage can be obtained during the test, or if pore pressure is not developed within the specimens during the test, this failure criterion is satisfactory.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] Head, Earth Materials Laboratory, United States Bureau of Reclamation, Denver, Colo.

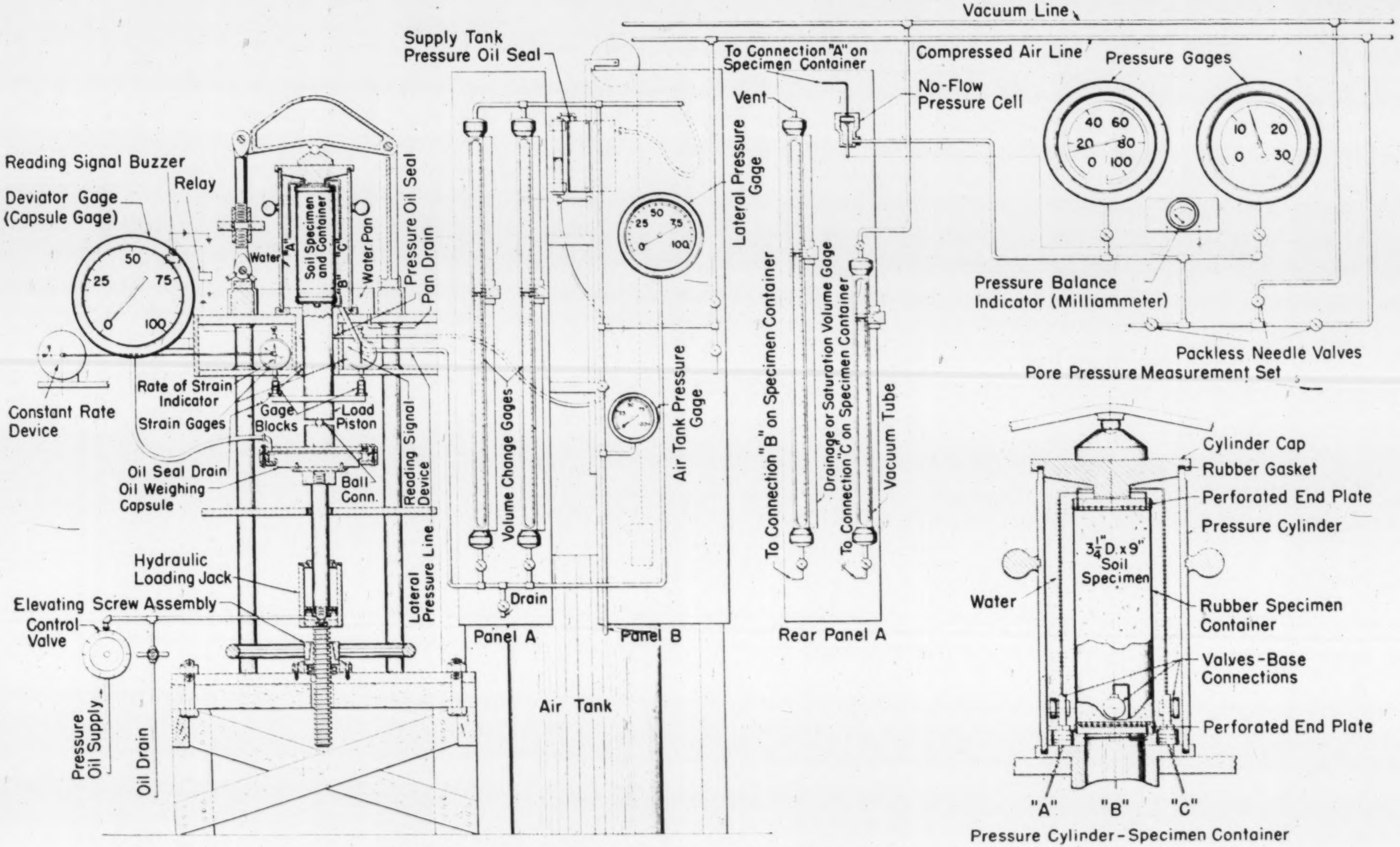
Vacuum Line
Compressed Air Line
Pressure Gages
To Connection "A" on Specimen Container
No-Flow Pressure Cell
Vent
Supply Tank Pressure Oil Seal
Reading Signal Buzzer
Relay
Deviator Gage (Capsule Gage)
Constant Rate Device
Rate of Strain Indicator
Strain Gages
Gage Blocks
Load Piston
Ball Conn.
Oil Seal Drain
Oil Weighing Capsule
Hydraulic Loading Jack
Elevating Screw Assembly
Control Valve
Pressure Oil Supply
Oil Drain
Water
"A"
"B"
"C"
Soil Specimen and Container
Water Pan
Pressure Oil Seal
Pan Drain
Reading Signal Device
Lateral Pressure Line
Volume Change Gages
Drain
Panel A
Panel B
Air Tank
Lateral Pressure Gage
Air Tank Pressure Gage
To Connection "B" on Specimen Container
Drainage or Saturation Volume Gage
To Connection "C" on Specimen Container
Vacuum Tube
Rear Panel A
Pressure Balance Indicator (Milliammeter)
Packless Needle Valves
Pore Pressure Measurement Set
Cylinder Cap
Rubber Gasket
Perforated End Plate
Pressure Cylinder
$3\frac{1}{4}$" D. x 9" Soil Specimen
Water
Rubber Specimen Container
Valves-Base Connections
Perforated End Plate
"A"
"B"
"C"
Pressure Cylinder-Specimen Container

FIG. 1.—Diagrammatic Drawing of the Triaxial Shear Machine.

However, when pore pressure exists within the specimens, the criterion is no longer applicable. On the basis of studies which are presented herein, the maximum principal stress ratio (maximum: major principal stress/minor principal stress, σ_1/σ_3) is proposed as the "true" failure criterion when pore pressure exists within the shear specimen.

A brief description of the testing equipment and test procedures used by the Bureau of Reclamation is included in this discussion to show how the test data were obtained. Comments relative to the effect of pore pressure on the failure of a soil mass are also given to show the stress conditions existing within the specimen during the triaxial shear test. These descriptions and comments are followed by a detailed discussion of the failure criterion.

TABLE I.—DESCRIPTION OF FIVE SOILS TESTED.

Sample	Percentage Smaller than 0.005 mm.	Percentage Smaller than 0.05 mm.	Percentage Passing ¼ in. Sieve	Liquid Limit	Plastic Limit	Classification
No. OOT-3	5	13	100	(Sandy)		Sandy, well-graded—hard, angular to round particles.
No. 3RX-43	14	39	100	23	15	Sand, well graded, with clay binder—hard, angular to round, particles.
No. OOT-8	23	69	100	27	19	Clay, lean, silty.
No. ATX-3	44	84	100	30	19	Clay—moderate to high plasticity, slightly silty.
No. ATX-1	58	88	100	38	24	Clay—very plastic and very compressible.

TESTING EQUIPMENT AND TEST PROCEDURE

Axial compression with lateral support is the fundamental principle of the triaxial shear test. The general procedure, as used by the Bureau of Reclamation, is to enclose a soil cylinder in a thin-wall rubber container, the rubber container being sealed at each end to rigid, perforated plates. The sealed specimen is placed in a pressure chamber, and constant lateral support is applied by fluid pressure over the entire surface of the specimen. The soil cylinder is loaded axially under the constant lateral support until failure occurs. This procedure is repeated for other lateral pressures, and a relationship is thus established between the lateral pressures and the axial pressures at failure. The pore pressure is measured at the end of each specimen during the test, and the applied loads (axial and lateral) are corrected for the pore pressure. This correction is necessary in order to determine the effective particle-contact pressures, because the pore pressure acts equally in all directions in opposition to the applied loads. End pore-pressure measurements are made, instead of insert measurements within the specimens, because the end method is more applicable to standard test procedures. Previous laboratory studies on pore-pressure measuring devices show that accurate measurments can be made at the end of the specmens, for the soils tested in this research program, provided the test procedures and testing equipment described herein are used.

Figure 1 is a diagrammatic drawing of the triaxial shear machine used in the Bureau of Reclamation Laboratories. The essential features of this machine are as follows:

(*a*) The cylindrical soil specimen is encased in a thin-wall rubber container and sealed to perforated metal end plates. The top end plate is connected by a small

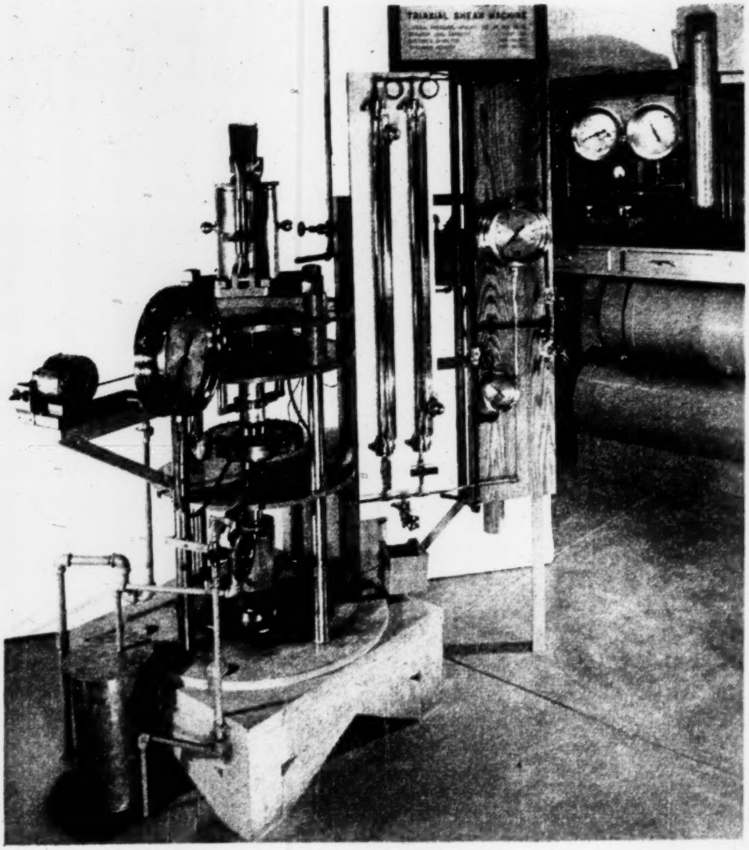

FIG. 2.—Triaxial Shear Machine.

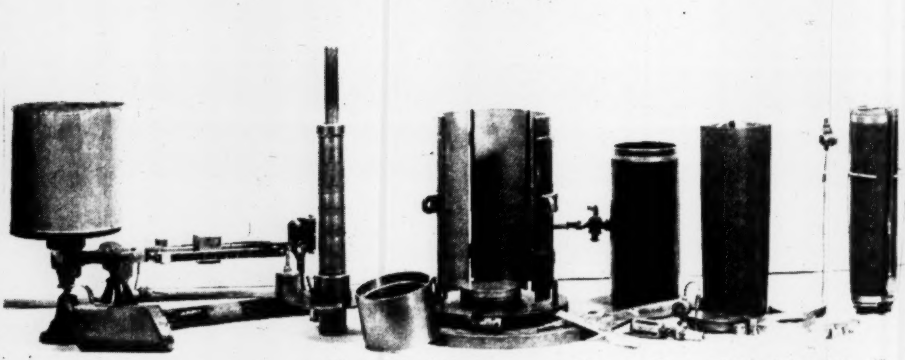

FIG. 3.—Preparation Equipment for Large Specimens.

(a) Triple beam balance.
(b) Compacting hammer.
(c) Compaction cylinder.
(d) Specimen trimming tool.
(e) Rubber container expander.
(f) Rubber container.
(g) Perforated top and bottom plates.
(h) Container clamps.
(i) Weighing carriage.

copper tube, filled with "air-free" water, that transmits pore pressure to a measuring device. A perforated bottom end plate may be used for obtaining drainage, if desired.

(*b*) The pressure cylinder is a separate unit that can be removed from the base. The cylinder with a top plate is clamped into position on a base plate by a framework controlled by a turnbuckle to form the pressure chamber around the soil specimen.

(*c*) The lateral pressure is applied to the water system surrounding the specimen inside the pressure chamber by means of air pressure from a large storage tank.

(*d*) The flow of water to or from the pressure chamber is measured by means of calibrated volume-change tubes and is interpreted as the volume change of the specimen.

(*e*) The axial load is applied by a hydraulic jack which is operated by nitrogen pressure over oil. The jack is manually controlled by a needle valve, and loads are applied to produce a constant rate of strain. The jack applies the load to an oil-weighing capsule, which, in turn, transmits the load to the loading piston.

(*f*) An oil-pressure seal around the piston prevents leakage of water past the bushing at the bottom of the pressure chamber.

(*g*) Two strain dials are used to measure the axial deformation by measuring the vertical movement of the piston into the pressure chamber. One strain dial is equipped with an indicator hand which is operated by an adjustable constant-speed device for use in controlling the load required to produce the desired rate of axial strain.

(*h*) The pore-pressure measuring equipment consists of a diaphragm-type pressure cell, a compressed air supply, gages for pressure measurements, and a milliammeter indicating device. The pressure cell is connected to the air-pressure supply on one side of the cell diaphragm. The other side of the diaphragm is connected by a small copper tube to the top perforated end plate of the specimen assembly, the system between the diaphragm and the specimen being completely filled with water. As the specimen is compressed, during the shear test, the pore pressure builds up and is transmitted to the diaphragm. The movement of the diaphragm makes an electrical contact with a stop, which is indicated on the milliammeter. Air pressure is then furnished to the other side of the diaphragm until the fluid pressure is equalled and the contact is broken.

Figure 2 is a photograph of the triaxial shear machine.

The Bureau uses the equipment in its present form for testing two sizes of specimens, $3\frac{1}{4}$ in. in diameter by 9 in. long and $1\frac{3}{8}$ in. in diameter by $2\frac{3}{4}$ in. long. The large specimens may either be remolded from loose earth materials or they may be cut from large undisturbed earth samples. The preparation equipment for molding large specimens is shown in Fig. 3. The small specimens are usually used when several specimens must be cut from undisturbed soil cores of 6-in. diameter. A small pressure capsule built nto a special pressure-chamber cap is used instead of the large pressure capsule for testing the small specimens.

For research work on remolded earth materials, a set of four companion specimens is usually tested. The specimens are controlled to a 0.5-lb. per cubic foot density and 0.1 per cent moisture-content range. After a specimen has been prepared and has been sealed in the container, a volume determination is made by weighing in air and water. The specimen is placed in the machine, and end-plate connections are made to the base receptacles for pore-pressure measurement, and for drainage, if desired. The pressure chamber is placed around the specimen and filled with air-free water, and constant lateral pressure is applied. An initial consolidation period is allowed. After consolidation under the lateral pressure is completed, the axial load is then applied at the constant rate desired until failure occurs. Generally, this rate is 0.05 and 0.01 in. of strain per minute for the large specimens and small specimens, respectively. These rates are slow enough so that the shear characteristics of the specimens are not affected. Volume change, pore pressure, axial stress, and axial strain measurements are taken at 2-min. intervals throughout the test period, the applied lateral pressure being held constant.

The test results are analyzed graphically by the use of the Mohr stress diagram, and the internal friction and cohesion values are determined from the envelope of limiting shearing resistance. The method of least squares is used to determine the most probable tangent to the stress circles in all research programs. For the construction of this diagram the effective lateral pressure is considered to be the minor principal stress (σ_3), and, likewise, the effective axial pressure to be the major principal stress (σ_1), where the effective pressures equal the applied pressures minus the measured pore pressure. It is assumed that the correction for pore pressure, in this diagram, allows the determination of the true shearing stresses which would exist in the absence of pore pressures. Tests with drainage are made only when the specimens can be completely drained throughout the test period; that is, when zero pore pressure conditions exist within the specimens. If complete drainage cannot be obtained, the specimens are tested in a sealed condition and the pore-pressure corrections are applied.

PORE PRESSURE

The effect of pore pressure on the shearing resistance of soils has been described in detail by other investigators,[2] and only a few brief comments are included in this paper. The shearing strength of a soil, from frictional resistance, depends upon the contact pressure between the soil grains. Pore-water pressure reduces these contact pressures and consequently reduces the shearing resistance. The void spaces of a soil mass are filled with a partially compressible fluid which consists of a mixture of gas and water, the compressibility of the mixture being dependent upon the relative proportions of gas and water. Loading a soil specimen in the shear test produces volume changes which must be accompanied by an extrusion of the pore fluid from the soil mass or by a compression of the fluid. The amount of pore pressure built up within the voids of the soil mass is dependent upon the loading conditions, the volume change characteristics of the soil, the amount of water in the soil voids, and the drainage characteristics of the soil.

Inasmuch as loading conditions imposed upon a laboratory test specimen are different from those imposed upon a soil mass in a structure, and, as the drainage restrictions may also be entirely different, it is our opinion that the reduction of the shear test data to zero pore-pressure conditions offers a method of analysis that is more easily interpreted and applied. In research programs where friction and cohesion are to be studied, the author feels that the reduction of the data to zero pore-pressure conditions is necessary to determine the "true" shear characteristics of the soil.

The results of 37 triaxial shear tests on impervious and semipervious soils in this

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[2] L. W. Hamilton, "The Effects of Internal Hydrostatic Pressure on the Shearing Resistance of Soils," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 1100 (1939).

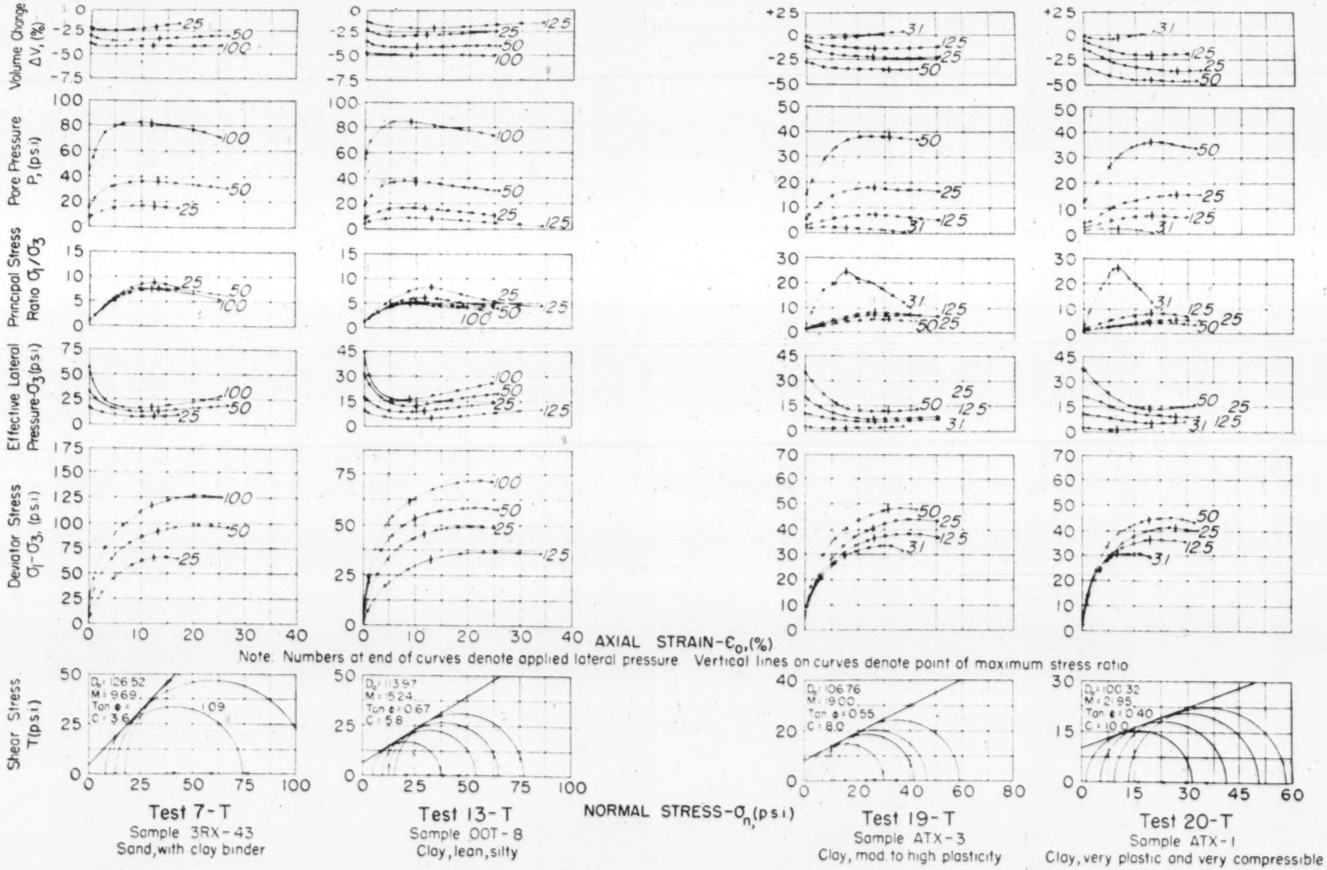

FIG. 4.—Triaxial Shear Tests on "Typical Materials."

research program, and numerous other shear tests which have been performed in connection with standard design programs, indicate that pore pressures are not constant during shear tests, although applied lateral pressures are held constant. Test results show that pore pressure builds up to some maximum value as the axial load is applied and then decreases as the axial load is increased. The diagrams of axial strain *versus* pore pressure, Fig. 4, illustrate these relationships for random moisture and density conditions on four of the five types of soils tested in this research program.

THE MAXIMUM σ_1/σ_3 FAILURE CRITERION

As previously indicated, when the research program was started the maximum deviator stress (maximum $\sigma_1 - \sigma_3$) was used as the failure criterion. Observations of pore pressure led us to believe that this criterion was true only as long as the effective lateral pressure (or minor principal stress, σ_3) remains constant. However, the effective lateral pressure does not remain constant although the applied lateral pressure is held constant. As discussed above, the pore pressure builds up to some maximum value as the axial load is applied and diminishes as loading is continued. Conversely, the effective lateral pressure, which equals the applied lateral pressure (a constant) minus the pore pressure, decreases as the axial load is applied to some minimum value, after which it increases as loading is continued.

It seems logical that, as the effective lateral pressure increases, because of decreased pore pressure, some gain in axial strength can be anticipated, even though failure might have already begun. That is, the axial strength should increase because the effective lateral support is increased. The maximum principal stress ratio appears to represent the most critical stress condition or the point of incipient failure under variable effective axial and lateral stresses. A study of the volume change conditions during the tests indicates that specimens consolidate to some minimum volume, after which the volume increases as loading is continued. It is believed that the minimum volume condition, or some point near this condition, indicates the condition of incipient failure. That is, the condition at which consolidation ceases and the mass begins to rupture. The maximum pore-pressure condition should occur when the specimen has been consolidated to a minimum volume, because at this point the pore fluid has been compressed to the greatest degree.

In correlating the test data, it was found that the point of maximum pore pressure, and minimum effective lateral pressure, coincided very closely with the minimum volume and maximum principal stress-ratio points. Examples of these relationships are shown by the data plotted in Fig. 4. The figure shows the plots of axial strain *versus* deviator stress, minor principal stress, principal stress ratio, pore pressure, and volume change throughout entire shear tests on four soils. The Mohr stress diagrams are also given on these plots. The initial density (D_0) and moisture content M for each test are indicated on the stress diagrams. Each shear test was performed under constant applied lateral pressure conditions. The lateral pressures used (in pounds per square inch) are indicated by numbers at the ends of the curves.

Figures 5 and 6 were plotted to show the stress conditions existing on the failure plane prior to, during, and after failure. Figure 5 will be recognized as conventional Mohr's stress-circle plots showing the circles for various stress conditions at different intervals of axial strain during the tests on two specimens (Nos. 9 and 1) of test 13-T on the "lean,

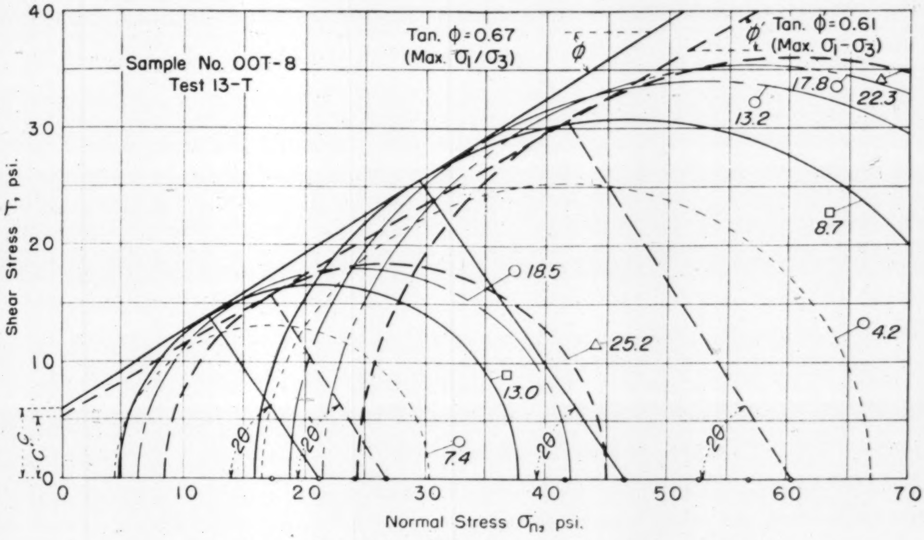

FIG. 5.—Mohr's Stress Circle Plots Showing σ_1/σ_3 Failure Criterion.

Note.—Test measurement intervals:

○ 12 Test measurement in percent of axial strain.
□ 12 Maximum principal stress ratio—(σ_1/σ_3).
△ 12 Maximum deviator stress—(σ_1/σ_3).

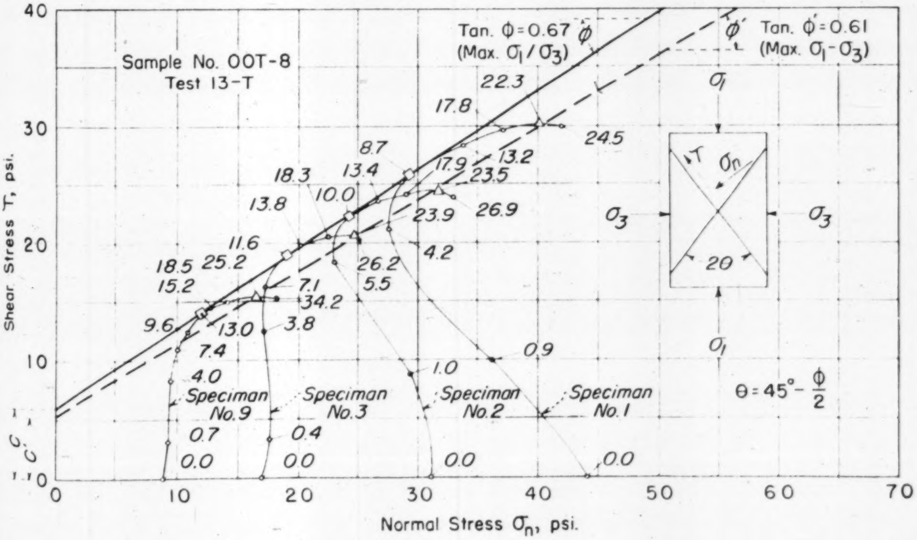

FIG. 6.—Shear and Normal Stress Conditions on the θ Failure Plane Showing Maximum σ_1/σ_3 Failure Criterion.

Note.—Test measurement intervals:

○ 12 Test measurement in percent of axial strain.
□ 12 Maximum principal stress ratio—(σ_1/σ_3).
△ 12 Maximum deviator stress—(σ_1/σ_3).

silty clay" soil. These specimens were tested under constant applied lateral pressures of 12.5 and 100.0 psi., respectively. The numbers opposite the stress circles indicate the degree of axial strain for which the stress data are plotted.

It can be seen by this diagram that, as the specimens are strained, the circles which represent the stress conditions for various degrees of strain increase in size as the deviator stress ($\sigma_1 - \sigma_3$) increases. The stress circles also move toward the y axis as the effective lateral pressure (σ_3) decreases, as a result of increased pore pressure. However, after the maximum principal stress-ratio point is reached, the stress circles move away from the y axis as the effective lateral pressure increases, as a result of decreased pore pressure. Accordingly, the stress circles which represent the stress conditions at the maximum principal stress ratio point are the "most effective" circles. These circles are shown by solid lines on the diagram (Fig. 5).

The stress conditions can be shown to greater advantage by the curves of Fig. 6 of the test data on all four specimens of series 13-T on the "lean, silty clay" soil. These specimens (Nos. 9, 3, 2, and 1) were tested under applied lateral pressures of 12.5, 25.0, 50.0, and 100.0 psi., respectively. This method of plotting, which has been used previously by D. W. Taylor,[3] clearly shows the stress conditions existing on the failure plane, during the entire triaxial shear test, for each of the four specimens. The diagram shows continuous curves of the intergranular stress components, normal stresses (σ_n) and shear stresses (τ), on the θ failure plane for all tests in this series. It will be noted on this plot that, for all specimens, the stresses at the "maximum principal stress ratio" represent the most critical stress conditions and further substantiate the use of the maximum principal stress ratio as the point of incipient failure.

SUMMARY

The reasons for the acceptance of the maximum principal stress-ratio point as the failure criterion for analyzing triaxial shear-test data are presented in the following summary:

1. When an impervious and semipervious soil is tested under sealed conditions at a constant applied lateral pressure, the pore pressure builds up to some maximum value as the axial load is increased, after which the pore pressure decreases as loading is continued.

2. Since the effective lateral stress equals the applied lateral pressure minus the pore pressure, the former decreases to some minimum value as the axial load is increased, after which this stress increases as loading is continued.

3. Shear specimens consolidate to some minimum volume as axial load is applied, after which the volume increases as the loading is continued. It is believed that this minimum volume condition indicates the point of incipient failure.

4. The maximum principal stress ratio, based on effective particle-contact pressures, represents the most critical stress condition or the point of incipient failure (see Fig. 6).

5. Within the accuracy of the test measurements, the points of maximum pore pressure, minimum volume, and maximum principal stress ratio coincide (see Fig. 4).

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[3] Tenth Progress Report to the U. S. Engineer Department, M.I.T. Soils Mechanics Laboratory, May, 1944.

DISCUSSION

MR. WILLIAM S. HOUSEL (*by letter*).[1]—It seems apparent to the writer that those engineers who are interested in shear testing procedures as a tool to be used in general engineering practice must be confused and possibly disappointed by what appears to be such divergent conclusions as are presented in the paper by Mr. Holtz and the paper of which the writer is co-author.[2]

In this connection it should be remembered that shear-testing technique as applied to soils has not yet emerged from the experimental stage as far as general practice is concerned. It is probably true that only those who are actually engaged in such research can appreciate the basic differences involved in the triaxial shear test and the direct shear or "ring shear" test. Even among those specialists there are many points of disagreement, and the use of shear test results in the solution of practical problems by those completely familiar with the tests must be savored with judgment and practical experience.

The writer would like to comment briefly on the essential difference in the two types of shear tests and thus attempt to clarify what might seem to be contradiction in the test results. The so-called "ring shear test" developed at the University of Michigan is a direct shear test conducted by applying transverse loads to a section of a cylindrical sample. As applied to purely cohesive soils such as plastic clay no axial load is used so that the normal pressure on the shear plane is zero. This procedure is followed for two reasons. First, it eliminates consolidation of the sample due to the applied pressure, thus avoiding changes in the specimen during testing which have long complicated the determination of the true shear strength of the original undisturbed material. In the second place, the testing at zero normal pressure is in agreement with the observation and analysis of the behavior of cohesive earth masses under natural conditions such as earthslides and displacement under large structures in which it appears that the actual shearing forces brought into action are independent of the normal pressure on the sliding surface.[3]

This behavior apparently means that such clay masses do behave as purely cohesive materials in which shearing resistance is independent of normal pressure.[4] It also seems apparent that the consolidation effects encountered in laboratory testing of an isolated sample are not realized in the large masses dealt with in nature.

These are the reasons for the procedure which has been used in the ring shear test, and while they have led to a successful correlation of shearing resistance with field observations on a large scale it must be realized that elimination of normal pressure components is only applicable to purely cohesive soils. In granular soils, shear resistance is due to internal friction and directly proportional to normal pressure. In this case consolidation of the sample and altered soil structure must be accepted as one of the complications to be mastered. The use of rapid shear tests, which are designed to measure the shearing resistance before consolidation takes place, encounter what is perhaps an even greater difficulty in the dynamic resistance to a rapidly applied load. For example, the type of rapid shear test used by the University of Michigan Laboratory for comparative purposes and conducted in parallel with the ring shear test consistently produces a shearing resistance in cohesive soils approximately four times the static yield value as measured in the ring shear test.

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[1] Associate Professor of Civil Engineering, University of Michigan, and Research Consultant, Michigan State Highway Dept., Ann Arbor, Mich.

[2] G. O. Kerkhoff and W. S. Housel, "Uplift Soil Pressure on Bridge Foundations as Revealed by Shear Tests," see p. 1056, this volume.

[3] K. Terzaghi, Unpublished discussion. See p. 445. of paper referred to in footnote 5.

[4] Symposium on Earth Pressure and Shearing Resistance of Plastic Clay (Paper No. 2200) *Transactions*, Am. Soc. Civil Engrs., Vol. 108 (1943); K. Terzaghi, p. 981; D. P. Krynine, pp. 1080, 1084; W. S. Housel, pp. 1038, 1104; R. B. Peck, p. 1101.

Some attempts to measure the increase in shearing resistance due to consolidation indicates that it is considerably greater than the internal friction itself.[5] Furthermore any accurate determination of the consolidation effects requires such an elaborate testing program and multiplicity of tests that it must be immediately ruled out as impracticable in any routine testing for actual construction projects.

Thus a shear testing procedure which might provide an accurate failure criterion for granular or semicohesive soils presents a very difficult problem. In the writer's opinion a sufficiently accurate method has not yet been developed. One thing that seems fairly certain is that such a method must provide for observation of the effect of the rate of loading and extrapolation of time effects to some common basis of comparison such as the yield value at a zero rate of deformation. Second, there must be some method of measuring the change in shearing resistance due to consolidation and the extrapolation of test results to the degree of consolidation found in the natural state.

In examining the validity of the failure criterion proposed by Mr. Holtz in the paper under discussion, the writer would first ask the question: Have the observations been made over a sufficient range of time variation and in such a manner that the time effects can be evaluated? It is felt that this question should be answered by demonstration rather than assumption.

The second question as to the validity of the criterion would be: Has the effect of volume changes or consolidation been so evaluated that the results may be extrapolated to any degree of consolidation that may be found under field conditions or in the undisturbed state?

After a more careful study of the paper since presentation at the meeting, the writer would like to comment on several other phases of the triaxial test as presented although it would be impossible to treat these matters exhaustively. A great deal of importance has been attached to the effect of pore water pressure in development of the maximum principal stress ratio as the failure criterion in evaluating shear test on earth materials.

In the analysis of the test results, the applied lateral and vertical pressures have both been "corrected" by the apparent pore pressure. The author also states that in previous investigations the "maximum deviatior stress" or in other words the maximum difference in principal pressures had been used as a failure criterion but "when pore pressure exists within the specimens the criterion is no longer applicable."

So far as the writer can determine the pore pressure correction of the pressures actually applied in the test and the statement that the maximum difference in principal pressures is no longer a valid criterion are both assumptions based on a certain behavior concept and are not demonstrated by the experimental data presented.

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[5] W. S. Housel, "Measurement of Cohesive Soil Properties Applied to Engineering Design," *Civil Engineering*, Am. Soc. Civil Engrs., Vol. 12, No. 8, August, 1942, p. 442.

In this connection the writer would emphasize that the use of the maximum difference in principal pressures as the failure criterion is in strict accordance with the accepted definition of a cohesive material and is the criterion used by the writer in determining the shearing resistance of clay soils by the ring shear test.

In discussing failure of any material under applied load it seems obvious that one must consider the applied stress and the resistance mobilized by the material in the relation of action to reaction which cannot be separated in any problem of static equilibrium. An equal reduction in both of the principal pressures due to pore pressure or any other factor has no effect on the applied shearing stress which is equal to one half the difference in principal pressures. As the load is applied the shearing resistance mobilized is equal and opposite to the shearing stress, and this equality does not vary even though the ratio of principal pressures is changing.

If it is assumed that the reaction of the material to stress or the shearing resistance is a function of internal friction and is proportional to normal pressure any decrease in such resistance due to the pore pressure correction must be accompanied by a proportional change in the applied shearing stress. As has been pointed out by the writer in discussing internal stability of granular materials the assumption of proportionality between principal pressures extends to normal pressure and frictional shearing resistance which with equality of applied stress and resistance mobilized by internal friction eliminates the possibility of stress exceeding resistance. To the writer this means elimination of a positive failure criterion under the conditions of static equilibrium.[6]

One could continue the discussion through the various ramifications involved in this highly controversial problem, but this would consume more space than should be taken up in this discussion. Much of such discussion would depend upon clarification of definitions and a definite decision would also require a detailed comparative analysis of the author's data which is also too involved for a discussion of the author's paper.

However, in closing the writer would like to point out that aside from the many complexities involved that the use of the maximum principal stress ratio as a failure criterion would be very difficult to distinguish from the use of the maximum difference in principal pressures by the method used by the author.

In a cohesive material subjected to triaxial load, this ratio would start out at a constant value and as a load is applied would increase up to the point that the shearing stress exceeded the shearing resistance. The ratio would then decrease, perhaps rapidly, if fracture or rearrangement of soil structure took place along the shear plane or fall off very slowly or not at all in very plastic soils if a constant rate of loading is maintained.

It seems to the writer, therefore, that the maximum difference in principal pressures and the maximum principal stress ratio would tend to be coincident, making it diffcult on the basis of test results to separate one from the other. It is on this point that the writer would particularly question the "effective lateral pressure" used by the author when the situation to which the material is subjected includes the complication of volume changes due to consolidation. Coincidence between the maximum pore pressure and the minimum volume followed by a decrease in pore pressure as the volume increases due to excessive deformation seems to be an obvious result. However the assumed increase in the "effective lateral pressure" is not so obvious. A free body diagram showing the external forces to which the sample is subjected throughout the loading process is sufficient demonstration for the writer that the constant applied lateral pressure is fully effective throughout the test.

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[6] W. S. Housel, "Internal Stability of Granular Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 465 (1936).

MR. L. A. PALMER[7] *(by letter)*. The Bureau of Reclamation and its reporter, Mr. Holtz, are to be commended for a splendid contribution in the field of soil testing.

A perusal of the paper brings several questions to the mind of this reader, many of which could best be discussed over the table. An important question that always arises in connection with shear test data obtained with clays is the utilization of the data. In most problems of foundation stability we are concerned with the shearing resistance of soil within a zone or region of shear that is at and near the elevation of the footing or applied load. Deep seated strata, even when soft, present serious problems of long-time settlement by consolidation, but they are not usually involved in typical foundation failures caused by lateral yield and consequent upheaval of supporting soil accidents which occur during construction or shortly thereafter. Since these occurrences are relatively not deep seated, they usually involve fine-grained soils which have not been consolidated by appreciable overburden. Consequently, prior to any effective degree of consolidation due to building load, the effective intergranular pressure normal to actual or potential surfaces of shear is necessarily small and for this reason, frictional resistance on these surfaces is also small. Cohesion is then about all that can be counted on to resist failure in shear of the supporting soil. The smaller the area over which footing load is applied, the more this is true. For this condition, the test of most value is the simple compression test, the cohesion being estimated as one-half the strength in compression of undisturbed cylindrical samples.

The author indicates that the reduction in volume of the sample subjected to a constant applied lateral pressure and a gradually built up vertical load is due to the compressibility of the air-water mixture in the voids of the soil. In this writer's experience there seems also to be some evidence of the escape of air from the sample during the test. An increase in the percentage saturation of the sample during test tends to indicate this condition. It is also possible that some of the air in the voids becomes dissolved in the water under considerable pressure. The reduction in volume followed by an increase in volume during failure as described in the paper fits the description of volume change behavior of triaxial samples tested in the laboratory of the Bureau of Yards and Docks. This Bureau has not thus far, however, attempted to measure the internal "pore pressure" of the samples.

This writer does not agree in general with the supposition that with gain in volume when failure of the sample has begun there can be a corresponding gain in axial strength even though there is an increase in the effective lateral pressure. This could possibly be true for samples in which frictional resistance is of considerably more importance than cohesion, such as sample No. 3RX-43 of the author's report. With essentially cohesive types of soils the cohesion is lost once the shearing surfaces are developed. In samples which are not actually fractured in shear, slip planes are developed, and with continued deformation and movement over these planes, cohesion may be supplanted by adhesion to an extent such that the shearing resistance subsequent to the development of slip planes never becomes zero.

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[7] Bureau of Yards and Docks, Navy Department, Washington, D. C.

In any relatively impervious soil that is continuously stressed while it is partially or totally confined, a certain moisture content, voids ratio, etc., compatible with a state of equilibrium, tends to be reached. Prior to the attainment of equilibrium by drainage, the fluid phase is subjected to stress. If the fluid cannot escape, then the neutral fluid stress becomes constant at all points within the sample, provided that the system of applied stresses (in this case vertical and lateral pressures) remains constant. This is one condition of equilibrium. The neutral, fluid stress is termed "pore pressure" regardless of the simple fact that the material may have no pores whatsoever as would be the case with a fat clay, pure kaolin, bentonite, etc., comprised of stacks of flat, scale-like particles encased in "envelopes" of water. In silt, mixtures of silt and sand, and in mixtures of clay, silt and sand, pores, no doubt, exist.

The thickness of the water envelopes covering clay particles is defined for each specific state of equilibrium. It may be diminished by either an increase in temperature without external load or by an external force or system of forces operating at constant temperature. It diminishes to an extent compatible with a new state of equilibrium. Some of the outer film, free water, is stripped off. In a confined sample with this stripped off film water free to escape, the sample "consolidates." In an oven the water escapes as vapor and the sample shrinks. A clay sample could be either consolidated or dried to the same shrinkage limit without any more pore pressure, acting as the driving force, in the one case than in the other. There is, however, a neutral, hydrodynamic stress in the fluid phase, the water envelopes of the clay, subjected to a system of external stresses, but this has nothing to do with pores or with pore pressure.

With the device described by the author, there exists a doubt in the mind of this writer as to whether it is pore pressure or soil pressure that is actually measured. If the soil is replaced with gelatin, cup grease or any other plastic material and subjected to external load causing it to flow or deform, would the pore pressure meter still register?

Pore pressure exists without doubt in all soil types except fat clays. Where pore pressure cells have been embedded in a large mass of soil in the field, there has been good evidence to support the belief that the pore pressure under these conditions can be estimated.

MR. F. C. ROGERS[8] (*by letter*).—Mr. Holtz has presented a remarkably concise and understandable picture of the triaxial-shear machine, test method, and data interpretation as used by the Bureau of Reclamation. It is indeed gratifying to note that the Bureau's equipment for this test has graduated from the make-shift, rubber-hose-pinchcock varieties used in so many laboratories (including the writer's). The sketch and picture shown in Figs. 1 and 2 of the paper should be an inspiration to those who feel that the usual triaxial testing devices can stand improvement.

The fact that pore pressure exists and also varies during the triaxial shear test is uncontradictable. Study of Figs. 4, 5, and 6 of Mr. Holtz's paper indicate conclusively that a true concept of soil structure (particle-contact) stress is impossible if pore pressure is ignored.

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[8] Professor of Civil Engineering, Rutgers University, New Brunswick, N. J.

Without pore pressure corrections, the trajectory of the shear *versus* normal stress curves, plotted in Fig. 6 cannot be predicted and the point of maximum principal stress ratio cannot be located. Mr. Holtz has justified the consideration of pore pressure, evaluated its significance, qualitatively and quantitatively, and, further, has devised a failure criterion which fully recognizes the significance of pore pressure.

Mr. Holtz has advised the writer that the triaxial shear tests performed in the Bureau's laboratories are aimed primarily at securing data for analysis of earth dams and foundations. In this instance it is necessary that the increase in shear strength concurrent with consolidation of the earth materials provide an adequate margin of safety over the shear developed in the soil by the gradually increasing weight of dam embankment. Hence, the shear and consolidation considerations are intimately associated, with density and pore pressure as correlating factors. The necessity for considering pore pressures is obvious.

The use of the triaxial test for estimating subgrade modulus and strength for the design of flexible pavements is rapidly gaining favor.[9] Undoubtedly, the intensity and variation of pore pressures are as real and as significant in tests performed for pavement design as in the application described by Mr. Holtz. However, the time-location-stress relationships of pore pressure in subgrades are unknown and, in applying the test results, the pore pressure would have to be forecast for uncertain conditions of loading (rate and intensity) and degrees of saturation. Since in the final application, the significance of pore pressure must be arbitrarily established, the need for complicating test equipment and procedure can be questioned. For pavement design the separation of particle-contact and pore pressure values in the triaxial test and the subsequent combining of the two for design may be circumvented, possibly, by using "undisturbed" specimens maintained as nearly as possible at natural voids ratio and adjusted to the most critical saturation condition.

The definition of the failure criterion as the maximum principal stress ratio, based on effective particle-contact pressures, appears to place the point of incipient failure at a deviator stress value less than maximum. Review of Fig. 4 indicates that the maximum pore pressure curves provide the only reliable guide for determining the strain at which the maximum principal stress ratio occurs and, consequently, definition of incipient failure by this method requires the evaluation of pore pressures during the test. However, it is noted that, for the four samples ranging from a sand, with clay binder, to a very plastic and compressible clay, the principal stress ratio is essentially maximum at an axial strain of 10 per cent, and the maximum pore pressure is close to 80 per cent of the applied lateral pressure. If these relationships are more than coincidence, it may be possible to establish a method for approximating the point of incipient failure and thereby avoid the complications introduced by pore pressure evaluation.

MR. D. W. TAYLOR.[10]—The soil engineers of the Bureau of Reclamation deserve much commendation for their pioneer work in pore pressure research. Data from such research permit greatly improved understanding of shearing characteristics of soils and in time should pave the way to improved methods of stability analysis. The writer emphatically subscribes to the belief that pore pressure determinations are of the greatest importance in shear research and in shear strength investigations and, generally speaking, agrees with the material presented in Mr. Holtz's paper.

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[9] "Design of Flexible Pavement Using the Triaxial Compression Test," *Bulletin No. 8*, Highway Research Board.

[10] Associate Professor of Soil Mechanics, Massachusetts Institute of Technology, Cambridge, Mass.

On certain details, however, and in particular on the idea furnishing the title of the paper, somewhat different concepts and interpretations are preferred to those given. The shearing stresses on critical planes, represented by the ordinates of points shown by square symbols in Fig. 6, are not believed to be shearing strengths, because the samples are able to withstand the larger shearing stresses that are attained at greater shearing strains and that are the ordinates of the points shown by triangles. On this basis the strength envelope, in the writer's opinion, is the dashed line through the triangular symbols rather than the solid line through square symbols.

Actually, however, neither of these envelopes appearing in Fig. 6 can be used in stability analysis work, because their abscissa is the intergranular pressure and the intergranular pressure at failure is usually an unknown quantity in any highly impervious soil mass that is being investigated for stability. This is still true if pore pressure measurements are made within the embankment, unless the observed pore pressures happen to be the values prevailing at the point of incipient failure; proof of this point is given by the pore pressure curves in Fig. 4, which demonstrate that the pore pressures at failure generally are not equal to the pore pressures in an embankment that has a margin of safety against failure. The type of strength envelope that must be used, in the writer's opinion, for expressing shearing strengths in a clay mass that is saturated or nearly saturated and that will not drain appreciably during shear is an envelope that expresses shearing strength as a function of the pressure to which the clay has been consolidated. The pressures to which a clay stratum is consolidated in nature can in most instances be estimated with reasonable accuracy.

The consolidation pressure thus is recognized as the most important variable affecting the strength in this case, but it must be realized that other variables also have effect. If the clay has been precompressed, the maximum pressure to which the clay has been consolidated in past ages is a variable that has considerable effect on the strength. If the soil is not completely saturated, the applied pressure has some effect on the strength. The factors that affect shearing strengths are not so well understood as they should be, but they form such a broad subject that a brief discussion would be of very little value.[11]

It is noted with interest that the author claims for the soils represented in this paper that satisfactory pore pressure measurements can be made at the ends of the specimens with the Reclamation Bureau procedures and equipment. It has been found by the writer that the triaxial equipment of the M. I. T. Soil Mechanics Laboratory gives greatly different pore pressures at the ends and at the center of completely saturated samples, such differences being entirely reasonable in view of the obviously different strains at these points. It surely is important to track down the reason for apparent contradictions of this type. If the differences are due to characteristics of apparatus, which is conceivable, but to the writer would not appear to be likely, such differences and their causes should be studied in detail. If satisfactory pore pressures determinations can be obtained at the ends of samples of certain types of soils and not in other types, it is of utmost importance that positive information be obtained as to which soil types fall into each category.

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[11] The writer has attempted to present a thorough discussion of this subject in "Fundamentals of Soil Mechanics," John Wiley and Sons, Inc., New York, N. Y. (1948).

The large amount of data assembled in Fig. 4 is an important addition to published information on shearing characteristics of clays. It is to be noted that these data are all for remolded soils, and the shearing strains represented are of the high order of magnitude that can normally be associated only with remolded soils. More data of this type should be published, presenting results for other soil types, and in particular for soils in the undisturbed state.

W. G. HOLTZ (*Author's closure by letter*).—The author wishes to express his thanks to the above writers for their efforts in discussing the subject paper. The author will attempt to present his views on these discussions, as presented by each writer, to clarify apparent differences in concepts and interpretations. It is regretted that discussions requested from E. J. Kilcawley and W. J. Turnbull are not yet available because of delayed invitations to them. Arrangements have been made to include their discussions in a future issue of the ASTM BULLETIN.

On comments by D. W. Taylor: In considering the shearing stresses on critical planes as represented by the square symbols in Fig. 6, it must be borne in mind that the author is talking in terms of "true" particle contact pressures in which no pore pressure is assumed to exist. Therefore, the fact that the test specimen (or soil mass) is able to withstand larger applied axial loads, because of reduced pore pressure, does not mean that actual failure has not begun. The plot of Fig. 6 clearly indicates where the normal stress is most effective in producing shear resistance. It will be noted that the internal friction values determined by the stress ratio criterion are larger than those determined by the stress difference criterion.

As indicated in the paper, one of the principal reasons for reducing all possible test results to zero pore pressure conditions is that this procedure offers a method of analysis that the author believes to be more easily interpreted and applied. For research study of the effects of such variables as moisture, density, etc., it is of great importance to observe the true point of incipient failure. The author recognizes the fact that the corrected values thus determined cannot be applied directly in design analyses. Anticipated pore pressures may be predicted on the bases of computations from consolidation data, or from pore pressure-consolidation tests (as made by the Bureau Laboratory), or perhaps be estimated from observations on existing structures. Such pore pressures (or uplift pressures) must then be taken into account in the determination of available frictional resistance. The author believes that two papers, recently prepared for Subcommittee 15 of the Second International Soil Conference on this subject, are of particular interest and clarify many present controversies regarding pore pressure development:

1. "Estimating Construction Pore Pressures in Rolled Earth Dams" by J. W. Hilf.[12]
2. "Ten Years of Pore Pressure Observations in Earth Dams by the Bureau of Reclamation" by W. W. Daehn,[12] and F. C. Walker.[12]

A great deal of effort has been expended by the Bureau Laboratory in perfecting equipment and techniques. The author believes such perfection is an absolute necessity, particularly in research testing. Most research and standard design testing requires the testing of a great number of soil specimens, and therefore the use of methods that are quick and efficient has been stressed. For this reason, particular attention has been given to the "end" method of pore pressure measurement. The author, however, recognizes the fact that end pore pressure measurements are difficult and sometimes impossible to obtain in highly saturated and highly impervious clays. The conclusions presented were therefore limited in scope to materials over the range shown. We believe we have made a step in the right direction, and the testing of highly impervious saturated clays will require a lot of additional hard research work. With this thought in mind, a small triaxial shear machine has been designed and is now under construction in the Bureau Laboratory shops. Remolded soils were used entirely in the subject program to attain adequate control over moisture, density, and composition. The data presented in the paper were only the partial results of the whole program which included tests at constant failure density, tests to determine the effect of moisture and density, tests on small *versus* large specimens, and tests on various pore pressure devices and inserts.

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[12] Civil Engineers, Office of the Chief Engineer, Bureau of Reclamation, Denver, Colorado.

One particular advantage of using the maximum principal stress ratio in determining the failure point for clay soils is that it is definite. Any investigator with experience in shearing clay soils will recognize the difficulties encountered in choosing the maximum deviator stress (σ_1-σ_3) point or the peak point of the deviator stress-strain curve. Some clays show almost constant deviator stress characteristics (or vary slightly up and down) over a wide range of axial strain. For this reason, better stress circle and envelope plots have been obtained, and many clay materials which we could not analyze heretofore give us straight line envelopes as shown in Fig. 4. Correct pore pressure correction is also necessary to obtain the straight line envelopes.

On comments by F. C. Rogers: The author recognizes the cost limits of developing triaxial shear testing equipment. A machine which will provide sufficiently accurate information for design purposes, compatible with pore pressure predictions and reliability of foundation or embankment samples, is all that is required from a design standpoint. The reason the Bureau of Reclamation has spent so much effort in developing its testing equipment is not only to get the best design data possible, but to further research in this field of soil mechanics. When dealing in terms of basic research, the best equipment available today may not suffice. We are therefore constantly striving to improve or redesign certain parts of the equipment to obtain the answers to questions constantly coming before us as a result of our studies.

The author believes that the relationship of the maximum principal stress ratio to 80 per cent pore pressure and 10 per cent strain is probably a coincidence due to the moisture content of the specimens represented in the paper. Higher or lower degrees of saturation would undoubtedly change the pore pressure at failure.

On comments by W. S. Housel: As indicated by Mr. Housel, there are still many points of difference existing among "specialists" involved in shear testing and the application of shear test results. The author has pointed out his beliefs regarding the use of corrected triaxial shear test data. There are methods in use for arriving at conclusions for for design purposes by the direct shear method. Some investigators using the triaxial shear test use "quick," "consolidated quick," and "consolidated slow" shear test data, as well as consolidation data, in arriving at conservative shear resistance values for design purposes. The author is not familiar with the testing techniques of the "rapid shear" test or the "ring shear" test used by Mr. Housel. However, it is generally true that when shear tests are made on unsealed specimens, the rapid test provides less specimen strength than the slow test because of the drainage of pore fluid that accompanies a slow test. It is recognized that consolidation produces higher shearing resistance because of the more intimate contact of the particles and the extrusion of pore fluid which must accompany consolidation. The Bureau Laboratory has spent considerable time in arriving at a suitable rate of testing. It is generally true that a rate of 0.05 and 0.01 in. of axial strain per minute is satisfactory for the large (3¼ by 9-in.) and small (1⅜ by 2¾-in.) specimens, respectively. Generally speaking, slower rates will not affect the strength. However, in testing highly saturated impervious soils, slower rates are usually necessary. The satisfactory rate in these cases can be determined by pilot tests. The rate of testing used has been verified many times by actual tests. The entire subject program consisted of 37 complete shear tests with an average of four specimens being sheared for each test. Other research testing, as well as continuous standard testing on hundreds of specimens, has given us a chance to observe these conditions. This is one of the advantages of the sealed triaxial shear test with pore pressure correction in that the rate of testing does not affect the strength under the conditions discussed above. The extrapolation of the change of strength due to consolidation is a separate problem as previously explained.

In commenting further on Mr. Housel's discussion, the author believed that he had presented sufficient data to bear out the conclusions regarding the maximum principal stress ratio criterion and that the conclusions reached were amply demonstrated by data rather than being mere assumptions. In fact, the criterion was arrived at after an analysis of the test data. The author does not follow along with Mr. Housel's conclusions based on the "free body analysis." His argument apparently is that pore pressure does not alter shearing stress, that shearing resistance mobilized is equal to shearing stress, and, therefore, pore pressure does not alter shearing resistance. He is undoubtedly correct in stating that shearing stress is not altered by pore pressure. All other conditions remaining equal, it is a recognized fact that the application of different load conditions to produce failure on a triaxial shear specimen of free draining sand (for a simple example) will not alter the failure angle θ. The author would like to point that the use of the suggested criterion is to determine the *actual point of incipient failure*, and thereby to determine the correct stress conditions producing failure and accordingly the correct failure angle.

On comments by L. A. Palmer: The simple compression test as indicated by Mr. Palmer, or better, the sealed triaxial shear test under zero applied lateral pressure, is perfectly acceptable for testing *completely* saturated impervious clay soils when effective particle contact pressures cannot be mobilized (100 per cent pore pressure conditions) because of extremely slow drainage of the pore fluid through the material itself or boundary materials.

Under the procedure used by the Bureau, no air can escape from the system during the sealed test. It is true that some air may escape to other parts (end plate or tubes) of the system and be replaced by water from these other parts, but the air content of the entire sealed system is not changed. As long as the air content of the entire system is not changed, no error from this cause can result. A study of the equipment (Fig. 1) will clarify this point. The percentage saturation will naturally increase during the shear test for, when consolidation takes place (the compressed air volume is less), the degree of saturation must become greater without any change in actual water content. Air is dissolved into water under pressure, but, for the pressures used, it is small, as determined by Henry's law. It may be difficult for one who has not observed and measured the effect of pore pressure to visualize the action taking place.

The author does not agree that high frictional resistance in comparison to cohesive resistance is required to support the conclusion that gain in volume indicates failure. Mr. Palmer states that in clay soils cohesion is lost once failure takes place. A study of any stress-strain curves on soft clays (flat curves) will refute this contention.

The author agrees that if the pore fluid cannot escape, the fluid pressures become constant under a system of constant applied stresses that are not critical. However, whether the term pore pressure must be confined to materials other than clays, because the clay particles cannot be stacked like marbles, seems to be diverging from the subject to that of nomenclature. Regardless of the stacking that may occur in fat clays, we know from experience that internal fluid pressures do exist in these clays, the magnitude depending upon the load conditions, consolidation characteristics, drainage conditions, and degree of saturation. The fact that clay soils exhibit frictional resistance proportional to normal load is demonstrated by Fig. 4. Free pore water should not be confused with film water.

Using the pore pressure measuring devices described by the author, pore fluid pressures and not soil pressures are measured. The pore pressure measuring device would measure the applied lateral pressure if the soil in the rubber sleeve were replaced by water or any other material having zero shear resistance. If a firm material, containing no permeable voids, were placed in the sleeve in place of the soil, no pore pressure could be measured. The pore pressure values obtained by our testing equipment and techniques can be checked directly by Boyle's and Henry's laws for the compressibility of air and the solubility of air in water, respectively, provided the air content of the specimen is not too small. When specimens with high degrees of saturation are tested, the air volume change is so extremely critical that our present volume change measuring devices are not sufficiently accurate to measure the volume change for pore pressure computation. This is true for clays as well as granular soils. It may be interesting to point out that pore pressure studies are not limited to soils. The pore pressures, built up in concrete specimens subjected to triaxial loading, are also being studied.

ROUND TABLE DISCUSSION ON WATER-FORMED DEPOSITS

INTRODUCTION*

By C. E. Imhoff[1]

The program for this session has evolved from the work of two sections of Subcommittee IV on Methods of Analysis of A.S.T.M. Committee D-19 on water for Industrial Uses. The first part of the program deals with the uses of the spectroscope in the chemical analysis of the constituents in water-formed deposits. The second part of the program is devoted to the problem of naming and writing chemical formulas for the various materials found in deposits.

In the process of making a quantitative chemical analysis, the analytical chemist must know beforehand what elements are present in the sample upon which the analysis is to be made. Knowing this, the procedures are varied or modified so that all of the elements known to be present are included in his analytical scheme. For example, if the analyst knows that the principal constituent is calcium carbonate, he will apply a simple limestone analytical procedure, whereas if he knows that the sample contains phosphorus, iron, and aluminum, the procedure he uses will be considerably more complicated than that of the simple limestone procedure. If in addition to this, he knows there are heavy elements such as lead, tin, and chromium, the analtyical scheme is further complicated because of procedures which are necessary for the separation and removal of these heavy metals. It is even possible that the analyst may entirely overlook the presence of some of these heavier elements and they will be erroneously reported as some other element.

The procedure for determining the major and minor constituents is often quite long, and the first paper on the program, given by Mr. Howard Jaffe, deals with the use of the spectroscope in the rapid determination of the metallic elements (cations) that are present. This instrument has been used widely by the U. S. Bureau of Mines in the study of many mineral ores.

The second part of the program deals with problems of nomenclature which have arisen within the A.S.T.M. groups using the polarizing microscope and the X-ray diffraction unit in the study of water-formed deposits. With the more common usage of these instruments to determine crystalline species, we are becoming more and more aware that these crystalline species are minerals whose constitution often cannot be represented best by chemical formulas such as are used for simple metathesis reactions. For example, magnesium silicate formerly was written as $MgSiO_3$, but since examination with special instruments has begun, it is now realized that magnesium silicate commonly forms as $3MgO \cdot 2SiO_2 \cdot 2H_2O$ and is identical to the mineral serpentine. Since many of these compounds are minerals, it is natural that these compounds should be described in the terminology used by the mineralogist. There is, however, lack of consistency and standardization among the water-treating people and even among mineralogists and chemists in general in regard to this problem of nomenclature. An outstanding example is that of precipitated calcium phosphate. The following list gives some of the names and formulas for precipitated calcium phosphate.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16-20, 1947.

[1] Water Conditioning Department, Allis-Chalmers Mfg. Co., Milwaukee, Wis.

Whitlockite
Tricalcium Phosphate
Apatite
Hydroxyapatite
Hydroxylapatite
Phosphorite
$Ca_3P_2O_8$
$Ca_3(PO_4)_2 \cdot H_2O$
$Ca_{10}(OH)_2(PO_4)_6$
$10CaO \cdot 3P_2O_5 \cdot H_2O$
$3Ca_3(PO_4)_2 \cdot Ca(OH)_2$

Much of the confusion is due to the controversial composition of calcium phosphate and this has further complicated the problem.

X-ray diffraction studies have shown that calcium phosphate gives an apatite pattern over a very wide range in composition which may differ considerably from any of the chemical formulas which may be written for precipitated calcium phosphate or calcium hydroxyapatite. There are two ways of spelling the hydroxy or hydroxyl form of the apatite. The term phosphorite has been used by some to denote that precipitate in which the calcium-phosphorus ratio approaches that given for the tri-calcium phosphate formula $Ca_3(PO_4)_2 \cdot H_2O$. Many mineralogists claim there should be no distinction as the compound is still hydroxyl apatite plus absorbed phosphate. In regard to the five different ways of writing the chemical formula for calcium phosphate, each formula has certain good reasons for the manner in which it is written and shows some properties not shown by the others.

Section C of Subcommittee IV has considered this problem of nomenclature and has suggested that the compounds found in water-formed deposits be divided into three main classes and that the rules for naming these divisions be as follows:

1. The chemical name and chemical formula will be used for a substance occurring in only one crystal form and having a simple chemical constitution which is not subject to controversy. Example: Sodium chloride, $NaCl$.
2. The mineral name and chemical formula will be used for a substance occurring in more than one crystal form and having a simple chemical constitution which is not subject to controversy. Example: Calcite, $\beta CaCO_3$ and Aragonite, γ $CaCO_3$.
3. The mineral name and mineral formula in terms of oxides will be used for a substance occurring in one or more crystal forms and having a complex chemical or structural constitution which is subject to controversy. Example: Analcite, $Na_2O \cdot Al_2O_3 \cdot 4SiO_2 \cdot 2H_2O$; Hydroxyapatite, $10CaO \cdot 3P_2O_5 \cdot H_2O$.

In order to set the background for the discussions which are part of this program, Mr. Michael Fleischer is presenting a paper giving the history of the nomenclature of compounds and some of the problems involved in the nomenclature system.

SOME PROBLEMS IN NOMENCLATURE IN MINERALOGY AND INORGANIC CHEMISTRY[1]*

By Michael Fleischer[2]

Synopsis

The purpose of this paper is to supply a background for the consideration of the nomenclature of the compounds found in studies of boiler scales, corrosion products, and similar deposits met with in industrial engineering practice. A brief historical review is given, in which the development of the nomenclatures of mineralogy and inorganic chemistry is sketched. Present practices in the naming of compounds in these fields are summarized. The problems presented by the phenomena of polymorphism and isomorphous substitution are discussed, with some recommendations. Past and present methods of classification of the naturally occurring silicates are given, with a discussion of the nomenclatures based on them.

Whenever facts, theories, or ideas in a particular field of knowledge are assembled, the need arises for a classification of the data and for a system of names to be used—the nomenclature of that field. This paper deals to a certain extent with classification, but is primarily concerned with nomenclature. Most of us take the system of names we use in our particular field of study very much for granted except when some flaw in that system causes us annoyance or difficulty. The specialization so characteristic in science today easily leads the scientist to become thoroughly accustomed to one particular specialized vocabulary. However, scientists often find that their work has carried them out of the little corner they have come to regard as their own into territories also claimed by other sciences, each of which has its own highly specialized vocabulary.

In this situation, it is not surprising that the scientist's reaction often is a mixture of dismay at the necessity of learning a new language and of irritation at its irrationalities, inconsistencies, and imperfections. However justified such a reaction may be, it needs to be overcome and replaced by a desire to make an examination of the nomenclatures involved, in the hope that they can be reconciled and improved.

This paper results from just such a situation. Those members of the Society who are concerned with water-formed deposits, scales, and corrosion products have found themselves dealing with artificial products identical with or similar to naturally occurring minerals. They have found themselves troubled by the different names used for these compounds by mineralogists and by inorganic chemists. This paper has been written, not to give answers to the specific problems that have arisen, but to furnish background material that may aid in understanding the origins of the differences that now exist in the applicable nomenclatures. In other words, it may be worth-while before deciding where we go from here to see how we got where we are.

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

[1] Published by permission of the Director, U. S. Geological Survey, Washington, D. C.

[2] U. S. Geological Survey, Department of the Interior, Washington, D. C.

PURPOSE AND NATURE OF NOMENCLATURE

The need for and aims of nomenclature in science were stated in 1787 by Lavoisier, one of the founders of modern chemical nomenclature:

> "In every physical science there are three things to consider: namely, the series of facts that constitute the science, the ideas that recall the facts, and the words that express them. The word should give birth to the idea, and the idea should portray the fact: these are three impressions of the same seal. Since it is the words that preserve the ideas and transmit them, it follows that it is impossible to improve the science without perfecting its language and no matter how true the facts may be, however correct the ideas born from them, they will transmit only false impressions if there are no exact expressions to convey them. The perfection of chemical nomenclature thus consists in rendering the ideas and facts in their exact truth, without suppressing anything they present, and, above all, without adding anything to them; the word must be nothing but a faithful mirror, for it cannot be repeated too often, it is neither nature nor the facts she presents but our own reason that deceives us." (As quoted by Oesper (**20**)[3].)

This may present an ideal to aim at. In practice, we find that once a branch of science has adopted and used a system of nomenclature, further efforts are directed chiefly to modifications made necessary by new facts and new viewpoints. Changes generally are made slowly and with difficulty and usually are agreed to only after the difficulties that have arisen become serious. One frequently hears or sees statements like the following (**25**)

> "Could we but wipe out all the existing names and start afresh, it would not be a very difficult task to create a logical system of nomenclature. . . .We want . . . in chemistry a nomenclature based on principles so simple that a few hours' study would enable a chemist to write the name or the formula of any chemical compound of known constitution. We have, however, to suffer for the sins of our forefathers in chemistry; it would be impossible to expunge from the literature all their inconsistencies; in fact, one of the first things agreed upon by the International Union of Chemistry was that interference with the existing nomenclature should be as little as possible."

The only part of the preceding quotation that I should accept without reservation is the statement that it is impossible to change nomenclature by wiping out what exists, and starting anew. Anyone who has tried to persuade a group to make even minor changes is aware of the resistance that is sure to be met, and of the danger of accomplishing nothing if too much is attempted. L. J. Spencer, an eminent British mineralogist, discussing changes in mineralogical nomenclature, says:

> "Anything that is done must be quite simple and in gradual and persuasive (give and take) steps. It is not the slightest use to propose any drastic changes." (Quoted in (**18**).)

Even though it is conceded to be impossible to wipe out all existing names and create a new, logical system of nomenclature, the implication that this would solve our problems if it only could be done is worth considering further. Even with a fresh start, the creation of a logical system of nomenclature might not be so easy until we had a language more consistently constructed than we now have. If we wanted permanence, we should have to prevent the meanings of words from changing as they now do. "A word is not a crystal, transparent and unchanged; it is the skin of a living thought and may vary greatly in color and content according to the circumstances and the time in which it is used." (Justice Oliver Wendell Holmes.) Also we should have to solve the difficulties caused by language differences.[4] However, it is probably true that a reasonably logical system of nomenclature could be set up for any given branch of science; in fact, several systems of nomenclature, each one logically following a particular concept, probably could be set up. It does not follow that any one of them would be satisfactory in actual use. For example, when the phlogiston-antiphlogiston controversy was at its height, logical systems of nomenclature in chemistry could be constructed by either party to the controversy, and the system eventually discarded was not necessarily less logical than the survivor. Similarly in mineralogy, rival systems of nomenclature competed in the early nineteenth century. These were primarily based on classification by chemical or by physical properties. The nomenclatures based on classification by physical properties have disappeared, even though they were far more rigorously logical in their construction.

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[3] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1107.

Remarks like those about "the sins of our forefathers in chemistry" and their "inconsistencies" seem to miss the point that the nomenclature of a growing and changing science cannot be static. Supposing a logical or even a satisfactory nomenclature for inorganic coordination compounds had been unanimously adopted just before Werner's theory was announced, how satisfactory would it have been a few years later? Another example, discussed further below, is the classification of the natural silicate minerals, based up to twenty years ago on the silicon-oxygen ratio, but now based on crystal structure. Such shifts in fundamental concepts usually necessitate changes in nomenclature. Many, if not most, of the alleged "sins of our forefathers" consist of their inability to predict accurately advances made in science many years or even generations after their time. I think the indictment should be dismissed.

We should, therefore, not aim for the impossible perfect nomenclature, but should try to improve what we have, making necessary compromises without qualms about perfection or logic. Three of the desirable attributes of usage to be adopted are uniformity, consistency, and brevity. It is frequently impossible to have all of them, and some advantage must often be sacrificed. Uniformity in usage seems to me to be the most important consideration; it is better that all use the same inadequate name for a compound than that two or more names, any one of which is satisfactory, be used for a single compound. Consistency, that is, naming analogous compounds in analogous ways, is obviously desirable. Brevity may not always be desirable; the use of trivial[5] names, even those free from false significance, is generally discouraged in chemistry. They remain with us; despite the efforts of those who insist that the name of a compound must recall its composition, scientists are human enough to use cupferron (a notably bad name) in place of nitrosophenylhydroxylamine ammonium salt.

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[4] These, greatly increased by the growing importance of the Russian literature, are not further discussed in this paper. See Seidell (**24**) and Spencer (**27**) for examples of these problems.

[5] We call a name trivial if it is one that is not produced by the use of a systematic procedure of naming, as contrasted with systematic names such as those used in zoology, etc. Ideally, two specialists in a field that uses systematic names would arrive at the same systematic name for a new species, whereas two specialists in mineralogy would very likely propose two entirely different and unrelated trivial names.

HISTORICAL REVIEW[6]

Many of the names used in mineralogy and in chemistry are very old—some so old that their origins are lost. Many names still in use were listed by Pliny in the first century A. D., most of them names of native or easily reduced elements, of important ore minerals, or of gem minerals. The natural sciences, the various branches of which were not yet distinguished, showed relatively small advances up to 1700. This was the age of alchemy; many chemical reactions were discovered and many compounds were prepared. One can scarcely speak of a nomenclature then in use, but some of the names then given—names like sweet spirits of niter and oil of vitriol—still linger on, though long ago discarded from systematic nomenclature.

In the eighteenth century, the sciences showed remarkable advances. The phlogiston theory ruled chemistry until late in the century, when quantitative experiments were first made. Lavoisier found that he needed a new nomenclature for his antiphlogiston chemistry. Fortunately it was at hand; de Morveau, with Lavoisier, Berthollet, and de Fourcroy collaborating, published his nomenclature in 1787. With the discarding of the phlogiston theory in the early nineteenth century also went the nomenclature based on it, and de Morveau's was generally adopted. It was basically the same as that of inorganic chemistry today, though many important modifications have been made. (See Oesper (**20**) and Muir (**19**).)

Advances were simultaneously made in mineralogy, and several systematic classifications were set up. These, however, were based on qualitative description. In the last years of the eighteenth century, the first attempt at accurate crystallographic measurements and the first quantitative chemical analyses were made. These led to two groups of classifications of minerals and to different nomenclatures based on them. One group was based on physical properties, most commonly on crystal habit, and also on hardness, luster, cleavage, etc.; the second group was based on chemical properties, the arrangement at first being by cations, but later by anions.

Today, of course, we still have varying arrangements of minerals for specific working purpose. Thus we have tables for identification of minerals in which they are arranged according to their optical or physical properties. These, however, differ from the rival classifications of the early nineteenth century, which represented differences in viewpoint as to what constituted the domain of mineralogy. Mohs and some of his followers insisted that the chemical composition of a mineral belonged to chemistry only. They excluded from mineralogy all properties that could not be determined without destroying the mineral. The so-called natural history classifications and the nomenclatures based on this concept were strongly influenced by the work of Linnaeus in the biological sciences. Elaborate systems of genera and orders, usually with Latin names, were set up, and the names used consisted of two, three, or more Latin words, which were so cumbersome that it was generally found desirable to use also a single-word trivial name.

Those mineralogists, like Cronstedt and Berzelius, who considered its chemical composition to be the most important property of a mineral, set up rival classifications and nomenclatures. Their names were also likely to be cumbersome, and they too used trivial names for minerals. Both groups were able to point to instances in which the opposing system lead to the juxtaposition of unlike minerals, and the separation of closely related minerals. The chemical classifications gained slowly, and, by about 1850, the natural history classifications with their elaborate Latin terminology had been generally discarded. However, although the classification of minerals today is still primarily chemical, subdivisions are made on the basis of crystal structure.

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[6] Those interested in more than this brief and sketchy outline should consult the references at the end of the paper, especially Chester (**5**, **6**) Kobell (**12**, **13**), and Kopp (**14**).

MINERAL NAMES

The most obvious difference between the nomenclatures of inorganic chemistry and mineralogy today is that the former uses systematic descriptive names, such as sodium chloride, almost exclusively, with relatively few trivial names,[5] the latter uses trivial names, such as halite, exclusively. The situation probably exists in part because, as we have seen, the main features of the present usage in inorganic chemistry were put forward in 1787 and were widely adopted not long thereafter, whereas general agreement of the best basis for mineralogical classification was not reached until fifty years later, by which time the trivial names were firmly entrenched in usage. Probably of equal importance was the fact that most of the compounds to be named in inorganic chemistry in the early part of the nineteenth century were fairly simple in composition and could be given simple names. This was not and is not true of many of the most common minerals. For example, a possible name for the mineral of formula $KAl_2(AlSi_3)O_{10}(OH)_2$ is potassium dihydroxy dialuminum aluminotrisilicate;[7] mineralogists may perhaps be pardoned for continuing to use the name muscovite.

Also, the pressure for the use of systematic in place of trivial names has never been very great in mineralogy, simply because the number of names is not overwhelmingly large. The exact number of mineral species depends on the definition used, but is probably about 1500 to 2000. There are also a few additional names, as of special varieties, such as ruby and emerald, whose use is necessary or justifiable. Furthermore, only about twenty new names are proposed each year, of which more than half are relegated into the synonymy. Thus, the burden of these names has not yet become so intolerable that the science has been forced to name minerals systematically. The time for such naming may come, but no one in recent years has suggested such a complete revision of mineralogical nomenclature.

How are minerals named? They are named as the namer wishes, with no more system than that the name usually, but not always, has the suffix *ite* added to it. It was formerly considered preferable to give names, usually derived from Greek or Latin words, that gave some information about the mineral, such as color (albite, white), crystal form or habit (acmite, a point, from its sharp-pointed crystals), specific gravity (barite, heavy), or chemical composition (calcite and zincite). The idea seems good, but actually there are too few distinctive properties to go around, so that many of the names could appropriately designate more than one mineral. We thus have (**28**) from the Persian word for blue the names azurite (a basic copper carbonate), lazulite (a magnesium aluminum phosphate), and lazurite (a sodium aluminum sulfosilicate), also called lapis-lazuli. We also have another set of names from the Greek word for blue, as kyanite (an aluminum silicate), and still another set of names from the Latin word for blue, as ceruleite (a copper aluminum arsenate). In spite of this, there are many blue minerals whose names give no indication in any language of the color (for example, covellite, boleite). Furthermore, the name chosen may not fit all varieties of the mineral, as for example, the variety of acmite that occurs in blunt crystals, or green or colorless kyanite. However, such minor discrepancies in signification are not too troublesome, especially nowadays when few of us have enough Greek and Latin to know what the name was intended to mean. If, however, a name is too misleading, it should be changed. An example is the name hydrokyanite which might reasonably be expected to be a hydrated aluminum silicate (hydrous kyanite), or at least a blue, hydrous mineral. It is actually neither; it is colorless, anhydrous copper sulfate, given its name because it turns blue when water is added to it. This name obviously should be changed.

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[7] This is given as a possible name, not as the preferred name. Several other names, equally complicated, are

These examples illustrate the possible disadvantage of giving a name that means something. This has been used as an argument in favor of names that have no special meaning, like the most popular names of minerals, those that are derived from the names of persons. This custom originated in 1783 and was violently opposed by some mineralogists, but it is so pleasant a way to compliment a friend that it continues. Most namers of minerals feel that the persons thus honored should be scientists, though not necessarily mineralogists, but the list of persons so honored also includes mineral collectors, mine owners and officials, and political figures. There is a willemite for William I of the Netherlands, and the very recently named rooseveltite for F. D. Roosevelt, and even a mussolinite, but somehow we escaped a hitlerite.

Many minerals are named for the first locality at which the mineral was found. Examples are anglesite, from Anglesea, England, aragonite, from Aragon, Spain, and strontianite, from Strontian, Scotland. Some opposition to these names was based on the ground that it was illogical to speak of piedmontite (that is, a mineral from Piedmont) from Pennsylvania, but this has not prevented the common use of such names.

Some of the mineralogists who published classifications of minerals, especially before 1850, had prejudices against one or another of these ways of naming minerals. A few carried their prejudices so far that they rejected all names of a particular kind, such as all names based on localities, and substituted others. As a result, there was a chaotic condition, with several names being used for a single mineral, which led Kobell (**12**) to remark, "When one looks at the nomenclature of mineralogy, one is involuntarily reminded of the Tower of Babel." This has been corrected for most minerals, very largely without action by official groups, but rather chiefly by decisions of individuals whose opinions were regarded as authoritative. The standard reference work in mineralogy in the United States has long been Dana's System of Mineralogy (**9**), and its decisions on nomenclature have been accepted almost completely in the United States and have been very influential in all other English-speaking countries.[8]

Enough discrepancies in usage remained to prompt the Mineralogical Society of America to appoint in 1921 a Committee on Nomenclature to study the situation. Two reports were published, but no definite action was taken, possibly because majority and minority reports were submitted. A few years later, in 1930, a new committee was appointed. No attempts were made to reach general international agreement, but meetings with a similar British committee resulted in agreement on many disputed points (**18**). What followed shows why reformers sometimes get discouraged. The unanimous report of the American committee was formally adopted by the Mineralogical Society of America, but the Society's own journal does not insist on the use of the names approved. The British Society has taken no action on the report of its committee. Nevertheless, the work of the two committees was not in vain; the recommendations made have been followed by most mineralogists, including the authors of the new seventh edition of Dana's System, and will probably become firmly established in the nomenclature of mineralogy.

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[8] J. D. Dana gives an interesting account of the principles he followed in Dana's System, 5th Ed., pp. XXIX–XXXIV (1868). This is reprinted in the 6th Ed., pp. XL–XLV (1892).

Names Used in Inorganic Chemistry

Much concern has been expressed for many years over the state of nomenclature in inorganic chemistry. Attempts at reform culminated in a report in 1940 by a committee of the International Union of Chemistry (**11**). This report is readily available, as are discussions of some of the problems involved (**8**, **23**), so only a few of the most pertinent points need be mentioned here.

In naming binary compounds, "the electropositive constituent should always be put first in the formula and in the name whenever it is recognizable from the character of the compound—as in salts or salt-like compounds. The name of the electronegative constituent is given the termination, *ide*" (**11**, p. 890). "The preceding rules which have been developed for binary compounds are valid, when suitably extended, also for compounds of more than two elements. Radicals having special names are treated like the elementary constituents of a compound when constructing names" (**11**, p. 892).

Examples: $NaCl$, sodium chloride
PbS, lead sulfide
$(NH_4)_2SO_4$, ammonium sulfate

A further rule is, "If several electropositive constituents are combined with one electronegative, then the rule holds that the more electropositive constituent is to be placed first, just as in the case of binary compounds" (**11**, p. 892).

Example: potassium calcium phosphate ($KCaPO_4$)

Here we begin to have difficulties, as it is not always known whether such compounds are double salts, as above, or coordination compounds, whose central atom is placed last in the name.

Example: potassium chloroplatinate (K_2PtCl_6), not potassium platinum chloride.

Increasing importance in nomenclature is now being assigned to crystal structure. As we shall see later in a discussion of the silicates, this has gone farther in mineralogy than in inorganic chemistry, but it is already important in the latter. Scott writes (**28**, p. 5869) of nomenclature from the viewpoint of indexing:

"Another criterion in selecting names for indexing is the representation of structure so far as it is known and so far as this can be done in line with general indexing policy. Since it is only in the last thirty years that the structure of the solid state has been investigated by X-ray and electron-diffraction studies, many of the older names for inorganic compounds are not now considered correct; if, however, they are firmly established, it has seemed unwise to attempt to change them for indexing. Where usage varies or is ambiguous or where new compounds are involved, the name selected for indexing is the one that best indicates the structure."

Possibly Scott has in mind situations like the following. The compounds $MgAl_2O_4$, Mg_2TiO_4, and Zn_2SnO_4 are called magnesium aluminate, magnesium titanate, and zinc stannate, respectively. All these compounds have crystal structures of the spinel type. If we write the formulas to show this, we have:

$MgAl_2O_4$
$TiMg_2O_4$
$SnZn_2O_4$

If the first is named magnesium aluminate, the names of the latter two should logically be titanium magnesiate and tin zincate. As it is known that no distinct anionic complexes are present, the best names might perhaps be magnesium dialuminum oxide, etc. At this time it seems better not to attempt to change the older names.

The report of the committee of the International Union (**11**, p. 890) recommends that, "Indication of the electrochemical valency in the names of compounds should be made only by Stock's method. This is done by means of Roman figures, placed in parentheses and following, without hyphen, immediately after the names of the elements to which they refer."

Examples: FeO, iron (II) oxide; Fe_2O_3, iron (III) oxide; but Al_2O_3, aluminum oxide, not aluminum (III) oxide, for elements with only one valence.

The report continues, "The system of valency indication by terminations such as -ous, -ic (ferrous, ferric) which was previously in use has proved unsatisfactory and should now be avoided not only in scientific but also in technical writing."

It remains to be seen whether this recommendation, involving the abandonment of a method long in use, will be followed by chemists. It is probably safe to predict that the change will not come easily.

Many ways have been used to indicate the proportions of the constituents in chemical compounds. The report states (**11**, p. 891):

"Indication of the stoichiometric composition should be made by means of Greek numerical prefixes which should precede, without hyphen, the constituent to which they refer. This method of showing the composition in names instead of by means of the valency is customary more especially with nonpolar compounds. It is also indicated in cases where the composition differs from that to be expected from the usual valency or in those in which the electrochemical valency is unknown. The prefix *mono* can generally be omitted. . . ."

Examples: FeS_2, iron disulfide
Co_3O_4, tricobalt tetroxide

The same method is also one of the two recommended for naming the salts of isopoly acids, that is, those acids that can be regarded as being formed from two or more molecules of one and the same oxygen-containing acid by elimination of water.

Examples: $Na_2B_4O_7$, disodium tetraborate
$Na_2Si_3O_7$, disodium trisilicate

It is evident that this system will give extremely complicated and unwieldy names for complex compounds, such as many minerals. The report recommends the use of the formula itself in such cases.

The alternative method recommended in the report for salts of isopoly acids consists of resolving the simplest formula into the component base anhydride and acid anhydride, then showing the basy anhydride-acid anhydride ratio be Arabic figures in parentheses.

Examples: $Na_2B_4O_7$ $Na_2O \cdot 2B_2O_3$, sodium (1:2) borate
$Na_2Si_3O_7$ $Na_2O \cdot 3SiO_2$, sodium (1:3) silicate

It is my opinion that the recommendation is a bad one, as it results in two incompatible names for a single compound. It seems to me to be bad to allow alternative names for $Na_2Si_3O_7$ to be disodium trisilicate and sodium (1:3) silicate, as these imply different sodium-silicon ratios. Furthermore, the method requires the useless operation of resolving the formula into its components. The once popular dualistic system of giving formulas in terms of oxides, though still widely used, is being abandoned in favor of formulas that represent the findings of crystal structure determinations. A recent remark by a soils scientist is pertinent, "Formulae employing integral numbers of oxide molecules are also attempted; mainly, one feels, as a polite gesture to the lingering ghost of Berzelius" (Marshall, (**16**)).

One wonders why this method of naming was not applied to the formula as written, instead of to the resolved formula. If $Na_2Si_3O_7$ had been written sodium (2:3) silicate, there would have been no confusion as to whether sodium or sodium oxide was meant. As the oxide formulation was recommended several years ago, it might cause even more confusion to change the basis used in naming compounds by means of Arabic figures. If so, the method had better be dropped completely.

NOMENCLATURE OF POLYMORPHOUS SUBSTANCES

The two foregoing sections have reviewed the general principles of nomenclature now in use in mineralogy and in inorganic chemistry. We turn now to the consideration of certain special problems common to both, which have not been specifically considered in most discussions of nomenclature.

Polymorphism is the ability of a compound to exist in two or more forms having different crystal structures but the same chemical composition. The phenomenon is very common; hundreds of polymorphous compounds are known, and many more will undoubtedly be found. As is well known, the physical properties of polymorphs may be very different, and their differentiation is frequently very important in technology.

The following methods have been used to name polymorphs:

1. In mineralogy, they are given different trivial names. These names usually are entirely independent of one another.

Examples: $CaCO_3$, calcite and aragonite
TiO_2, rutile, anatase, and brookite
Al_2SiO_5, andalusite, sillimanite, and kyanite.

Occasionally the name of a dimorph is made by using the prefix *para-:*

Example: $FeSO_4(OH)\cdot 2H_2O$, butlerite and parabutlerite

2. Polymorphous forms are sometimes distinguished by prefixing different Greek letters to the formula.

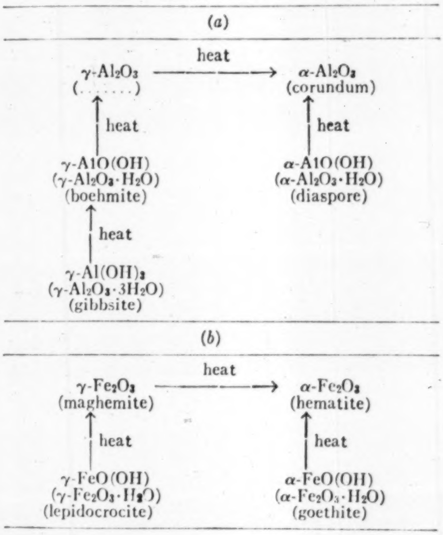
Examples: α-Fe_2O_3 (= hematite)
γ-Fe_2O_3 (= maghemite)
α-$AlO(OH)$ or α-$Al_2O_3\cdot H_2O$ (= diaspore)
γ-$AlO(OH)$ or γ-$Al_2O_3\cdot H_2O$ (= boehmite)

There has not always been uniformity in usage as to which Greek letter prefix applies to which polymorphous form, nor has there been uniformity in the way the prefixes have been used for polymorphs of different thermal stability. Most frequently, the form stable at the highest temperature is designated as the α-form, but this custom is not universal and the different forms may have the prefixes assigned in the order of their discovery. Once this has been done, later changes are extremely confusing.

There is no doubt that the use of Greek letter prefixes, when consistently applied, eases the burden on the memory.

This is well illustrated by Table I, which shows the changes that occur when aluminum and iron (III) oxide hydrates or hydroxides are heated. The analogy in behavior of the two series of compounds is much more easily remembered when the Greek letter prefixes are used. Unfortunately, however, there are few examples showing such consistency. For example, the forms of Mn_2O_3 designated as α-Mn_2O_3 and γ-Mn_2O_3 apparently are not analogous to the aluminum and iron (III) compounds. The two known forms of MnO(OH), one of which seems to be analogous to α-AlO(OH), have not yet been assigned Greek letter prefixes.

TABLE I.—CHANGES OCCURRING WHEN HYDRATED OXIDES OF ALUMINUM (*a*) AND IRON (III) (*b*) ARE HEATED.



3. In inorganic chemistry, polymorphous forms are less commonly distinguished by means of Roman numerals following the formula.

Example: Sodium sulfate, Na_2SO_4 I, Na_2SO_4 II, etc. to Na_2SO_4 V

The numerals have been applied by different scientists to different forms (*a*) in order of discovery, (*b*) systematically in order of stability, with the polymorph stable at the highest temperature designated as I, and (*c*) systematically in order of stability, with the polymorph stable at the lowest temperature designated as I. The use of (*b*) and (*c*) requires complete knowledge of the stability relations; the discovery of a new polymorph of intermediate stability requires either abandoning the systematic numbering or renumbering. The latter causes confusion as to just which form is meant.

Furthermore, although one can number the polymorphs systematically if one considers the changes with temperature at a fixed pressure (the most familiar case), or with pressure at a fixed temperature, no one seems to have proposed a system for numbering when both pressure and temperature variations are studied. The difficulty of getting a satisfactory answer to this problem is apparent from inspection of a few phase equilibrium diagrams, such as those given by Bridgman (**4**, pp. 232–233).

Polymorphs may be named in other ways, as by the use of qualifying words, such as high- or low-. Sometimes combinations of different methods are used. An interesting example is the nomenclature of the polymorphs of silica, SiO_2. The three most common forms that occur as minerals are named quartz, tridymite, and cristobalite. Each of these, however, undergoes with changes in temperture one or more inversions with the formation of new polymorphs. The resulting confusion is described by Sosman (**26**, p. 43):

> "When it was discovered that quartz has an inversion point at 573 C. at which its symmetry and properties change reversibly, the two modifications were distinguished by means of Greek-letter prefixes, the low-temperature modification being named α-quartz (alpha-quartz) and the high temperature modification, β-quartz (beta-quartz).

"Similar inversions in tridymite and cristobalite were later recognized, and the modifications were designated by prefixes corresponding to those used for quartz, namely, α- for the low-temperature modification, and β- for the high. When two high-temperature modifications of tridymite were found, the lower was called β_1, and the higher, β_2.

"Insisting too much on logic and too little on expediency, some of the German investigators (Boeke, Nacken, Rinne, and others) have within the past few years arbitrarily reversed the previously accepted notation, and now call the high-temperature modification of quartz α, and the low-temperature modification, β. They have likewise reversed the designations of the two forms of cristobalite, and, furthermore, have called α, β_1, and β_2 tridymite respectively γ, β, and α (gamma, beta, and alpha). These changes, if immediately and generally accepted, would bring the nomenclature into accord with the custom which now prevails generally, of naming the modification which is stable at the highest temperature α, the next lower, β, etc. In actual practice the changes have introduced confusion into the current literature on silica—that very worst type of confusion which consists in the interchanging of A and B or of + and −.

"I propose to avoid confusion by calling the modifications 'high-quartz' and 'low-quartz'; 'high-cristobalite' and 'low-cristobalite'; 'upper high-tridymite,' 'lower high-tridymite' and 'low-tridymite,' these terms being abbreviated forms of 'high-temperature quartz,' etc. Although less compact than the alpha-beta terms, these designations leave no doubt as to what is meant, and, incidentally, relieve the typesetter and proof-reader of some of their worries."

Despite such confused and awkward nomenclature, little has yet been done to systematize or unify usage in naming polymorphous forms. The need has increased as X-ray study has led to the discovery of many new polymorphs. It is certain that many more polymorphs will be found, and it is probable that more examples will be found of the peculiar type of polymorphism shown by silicon carbide. This compound is known as a cubic modification, β-SiC, and has at least seven hexagonal or rhombohedral modifications which have very closely related crystal structures. These last have previously been designated α-SiC, type I, and so on to α-SiC, type VI, in order of discovery. Very recently, Ramsdell (**21**) has suggested a new nomenclature for these compounds, based on the symmetry and on the number of layers in the unit cell. For example, one modification is silicon carbide, type 4H (H = hexagonal), another is silicon carbide, type 51R (R = rhombohedral). Serious consideration should be given to the problem of whether this method or others can be generalized.

PROBLEMS OF NOMENCLATURE DUE TO ISOMORPHOUS SUBSTITUTION

Isomorphous substitution or solid solution is of importance to inorganic chemists, as shown by the many experimental studies devoted to the subject. Yet little consideration has been given to the nomenclature of inorganic chemical compound showing isomorphous substitution. Perhaps this is because the inorganic chemist can and often does control his product to minimize isomorphous substitution. The mineralogist must deal with minerals as they are, and there are very few minerals that do not show isomorphous substitutions. Mineralogists have therefore been very much concerned about the nomenclature of varieties of minerals.

Let us consider first the simplest type of isomorphism, that in which there is replacement of one element by another of the same valence without change in the type of crystal structure, although there will be variations in physical properties such as specific gravity, indices of refraction, unit cell volume, etc. In this type of isomorphism, we may have a complete series of solid solutions, as in the alum series $KAl(SO_4)_2 \cdot 12H_2O$-$(NH_4)_2Al(SO_4)_2 \cdot 12H_2O$ or the olivine series Mg_2SiO_4-Fe_2SiO_4, or we may have limited solid solution, as in sphalerite (Zn, Fe)S, whose iron content ranges up to 26 per cent by weight.

How is one to name a compound that shows isomorphous substitution? In the past, this has been done in many different ways by mineralogists. Thus, magnesite containing $FeCO_3$ in solid solution might be written $(Mg,Fe)CO_3$ or $(Mg_{0.8}Fe_{0.2})CO_3$ if one fifth of the magnesium atoms are replaced by iron, and might be called ferriferous magnesite or iron-bearing magnesite or by the special name breunnerite. Special names have been given to dozens of varieties on the basis of isomorphous substitution. They are an unnecessary burden on the science and create special difficulties in indexing. Some names have been formed by running words together, as manganocalcite for a variety of calcite containing a little manganese. This is a term of dubious meaning; it could refer equally well to the manganese analogue of calcite, that is, to $MnCO_3$, rhodochrosite. Such terms have been used to mean partial isomorphous replacement, as in manganapatite and calciocelestite, and also to refer to analogous compounds, as jarosite, $KFe_3(SO_4)_2(OH)_6$, and argentojarosite, $AgFe_3(SO_4)_2(OH)_6$.

A great advance in naming these compounds was the proposal by Schaller (**22**) in 1930 of a uniform system of naming such varieties by means of adjectival modifiers. These are formed by adding the suffix *-an* (or *-ian* or *-oan* to represent different valences) to the name of the substituting element. In the example above, magnesite containing $FeCO_3$ is referred to as ferroan magnesite, and is indexed as magnesite, ferroan. The name breunnerite is discarded.

This method was opposed by some mineralogists as being too artificial a way of forming words, but its advantages have slowly convinced most mineralogists of its worth. It was recommended by the Committee on Nomenclature of the Mineralogical Society of America, has been adopted in the Seventh Edition of Dana's System, and is being used by some British mineralogists. Its general adoption may lead to the gradual disappearance of many special varietal names.

A few solid solution series have been given whole sets of special names for different compositional ranges. For example, six names have been given to different compositional ranges in the important olivine series, Mg_2SiO_4-Fe_2SiO_4. It is simpler and more satisfactory to characterize members of this series in terms of Mg_2SiO_4, forsterite (*Fo*), and Fe_2SiO_4, fayalite (*Fa*). Thus, we may write olivine ($Fo_{65}Fa_{35}$) or more simply olivine (Fo_{65}), that is, olivine containing 65 molecular per cent of Mg_2SiO_4. This is the common formulation in mineralogy and petrology. The equivalent formulation, $(Mg_{1.30}Fe_{0.70})SiO_4$, given in terms of atomic equivalents, is very much to be preferred.

The special names formerly much used for designating such intermediate members of series are used less and less, with only a few exceptions such as the names for the plagioclase feldspars. It is doubtful whether the use of more than two names is justifiable. A series such as $MgCO_3$-$FeCO_3$ is sufficiently characterized by the terms magnesian siderite and ferroan magnesite, according to whether iron or magnesium predominates, and by the formula, written $(Mg_x, Fe_{1-x})CO_3$.

There is no simple systematic way of naming compounds showing two or more simultaneous isomorphous substitutions. In the spinel group of minerals, which has the general formula AB_2O_4, A may be Mg, Fe^{II}, Zn, Mn^{II}, and other elements, and B may be Al, Fe^{III}, Cr, and Mn^{III}. An accurate representation of the composition of an actual mineral of the group can best be had by writing the formula to show the relative amounts present in terms of atomic equivalents, as $(Fe_{0.75}Mg_{0.25})\ (Al_{1.40}Fe_{0.38}Mn_{0.22})O_4$. This is the mineral hercynite; the particular sample might be called magnesian hercynite or magnesian ferrian hercynite, although in the original proposal by Schaller (**22**) the use of only one adjectival modifier was suggested. As we shall see later, such names are not very satisfactory for the complex silicates, and the use of formulas like that above seems preferable.

TABLE II.—SOME OF THE SUBSTITUTIONS KNOWN TO OCCUR IN THE APATITE STRUCTURE.

Substitution	Name
No. 1. $Ca_6(PO_4)_3F$ (end member)	Fluorapatite
No. 2. $Ca_6(PO_4)_3Cl$ (end member)	Chlorapatite
No. 3. $Ca_5(PO_4)_3(OH)$ (end member)	Hydroxylapatite
Mn for Ca[a] (in 1)	Manganapatite (better, manganoan fluorapatite)
Ce for Ca[a] (in 1)	Cerapatite (better, cerian fluorapatite)
Na for Ca[a] (in 3)	Dehrnite (better, sodian hydroxylapatite)
Sr for Ca[a] (in 1)	Saamite (better, strontian fluorapatite)
Sr for Ca[a] and As for P[a] (in 1)	Fermorite (better, strontian arsenatian fluorapatite)
Si and S for P[a] (in 1)	Sulfate-apatite, wilkeite (better, sulfatian silicatian fluorapatite)

[a] In each case, the subsitutions refer to partial substitution, that is, less than half of the element mentioned is replaced.

Anionic substitution is also possible, although perhaps less common, and can be treated by an extension of Schaller's adjectival modifiers. Table II shows some of the known varieties of the apatite mineral group, which seems to have a structure that can tolerate an unusual number of substitutions.

If the nomenclature of compounds showing isomorphism of the simple type just discussed is not entirely satisfactory, the situation is far worse for the many complex isomorphous substitutions that occur in minerals. Their existence has long been known and the substitutions for the plagioclase feldspars were even correctly formulated eighty years ago by Tschermak, but the general principles were established only by X-ray crystal structure studies in the decade from 1920 to 1930. Briefly, these principles are as follows.[9]

Most minerals are ionic in character. The decisive factor in the possibility of isomorphous substitution is the volumes of the ions in question without regard to their electrochemical valencies. An ion may be replaced by another ion of different electrochemical valence provided that another simultaneous substitution or substitutions balances the electrochemical neutrality of the assemblage. Thus, for example, sodium is replaced by calcium in the plagioclase feldspar series and simultaneously silicon is replaced by aluminum, and we have the complete solid solution series:

$$NaAlSiSi_2O_8 \text{ (albite) to}$$
$$CaAlAlSi_2O_8 \text{ (anorthite)}$$

Differences in ionic radii explain the lack of isomorphism between some ions of the same valence, such as calcium and magnesium. Different structures may permit more or less leeway in the sizes of substituting ions that can be introduced without changing the crystal structure, so that only the rough generalization has been made that most isomorphous substitutions are limited to ions whose ionic radii differ by not more than about 15 per cent. It must also be remembered that the ionic radii usually listed (**29**, pp. 417–419, and Dana's System, 7th Ed., pp. 4–5) are approximate and that the apparent ionic radii are affected by such things as the coordination of the ion in a particular structure and by its polarization.

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[9] **These are treated at length by Berman (1), Bragg (3), Stillwell (29), Evans (8), and Wells (32).**

However, we now have a basis on which to explain and predict isomorphous substitutions. If we consider the ionic radii (all in Ångström units) Ca 1.06, Mn 0.91, Fe 0.83, it is not surprising that $MnCO_3$ forms complete series of solid solutions with both $CaCO_3$ and $FeCO_3$, whereas $CaCO_3$ and $FeCO_3$ form very limited solid solutions. Likewise, the formerly mysterious fact that there were complete solid solution series between $NaAlSi_3O_8$ and $CaAl_2Si_2O_8$ and between $KAlSi_3O_8$ and $BaAl_2Si_2O_8$ but very little solid solution between the two alkali or the two alkali-earth silicates becomes reasonable when we consider the ionic radii: Na 0.98, Ca 1.06, K 1.33, Ba 1.43.

TABLE III.—CLASSIFICATION OF SILICATES BY OXYGEN RATIO METHOD.

Name	Oxygen Ratio	Example
Subsilicates........	Less than 1:1	Andalusite, Al_2SiO_5 ($Al_2O_3 \cdot SiO_2$)
Unisilicates[a].......	1:1	Forsterite, Mg_2SiO_4 ($2MgO \cdot SiO_2$)
Bisilicates.........	2:1	Enstatite, $MgSiO_3$ ($MgO \cdot SiO_2$)
Trisilicates........	3:1	Orthoclase, $KAlSi_3O_8$ ($K_2O \cdot Al_2O_3 \cdot 6SiO_2$)

[a] The term singulosilicate appears to have the same meaning as unisilicate.

Returning to our problem, how shall we name compounds that show complex isomorphous substitution? The most satisfactory way at present is to use a group name like amphibole and give the formula derived from the actual analysis in terms of atomic equivalents, as, for example,

$$(Ca_{1.92}Na_{0.67}K_{0.30})\ (Mg_{4.44}Fe_{0.10}Mn_{0.04}Ti_{0.12}Al_{0.35})\ (Al_{1.53}Si_{6.47})O_{22}(OH)_{0.63}F_{1.21}.$$

We now have a great many names for the members of complex groups such as the micas or the amphiboles. Some of the names are certainly superfluous. What we now need is a thorough compilation of the analyses, recalculation to formulas like that above, and classification. If we consider all the analyses conforming to a given type formula, for example $X_3Y_5Z_8O_{22}(OH, F)_2$ above, it would probably be justifiable to use a name for each predominant ion in each of the X and Y, and possibly in the Z positions for each symmetry type, but to discard names given for minor substitutions. This formidable task will confront the editors of the Seventh Edition of Dana's System when they begin to work on the silicate minerals.

TABLE IV.—CLASSIFICATION OF SILICATES AS SALTS OF HYPOTHETICAL SILICIC ACIDS.

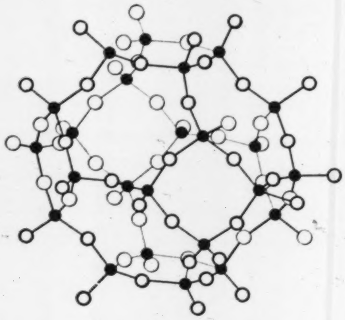
Name	Hypothetical Acid
Orthosilicates..........................	H_4SiO_4
Orthodisilicates........................	$H_6Si_2O_7$
Metasilicates..........................	H_2SiO_3
Metadisilicates........................	$H_2Si_2O_5$
Trisilicates or Polysilicates.............	$H_4Si_3O_8$

They will have the sympathy of all mineralogists in their task, and their conclusions very likely will have the disapprobation of a good many, no matter what those conclusions are.

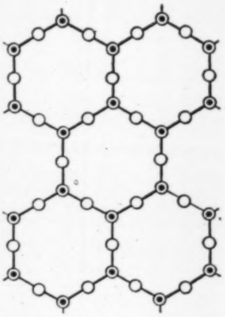
CLASSIFICATION OF THE NATURAL SILICATES

The silicates are among the most important and most complex minerals. Their constitution was studied and debated for many decades. Elaborate constitutional formulas were proposed, based on various chemical and physical properties. These are now of historic interest only; they were reviewed at length by Clarke (**7**) and more briefly by Mellor (**17**), and need not be discussed here. The determination of crystal structures by X-ray studies has finally led to an understanding of the constitution of the silicates, and the older theories have had to be discarded. Their impress on nomenclature has remained, however, and needs some mention.

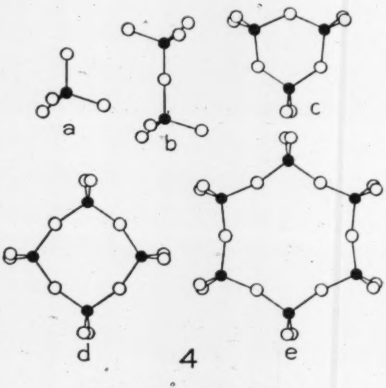
One of the methods formerly used to classify silicates was based on the ratio of oxygen atoms attached to silicon to oxygen atoms attached to bases. These are illustrated in Table III. Some of these terms are still in use, particularly by metallurgists in discussions of slags.

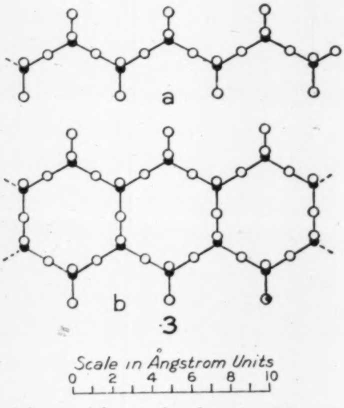


1



2





Linkages of silicon-oxygen tetrahedra. Black, silicon, with or without aluminum; white, oxygen. After Bragg.

FIG. 1.—Type F, three-dimensional network structure, SiO_2.

FIG. 2.—Type E, two-dimensional sheet structure, $(Si_2O_5)^{-2}$

FIG. 3.—Type D, chain structures.

(*a*) single—chain linkage $(SiO_3)^{-2}$. (*b*) double—chain linkage $(Si_4O_{11})^{-6}$.

FIG. 4 (*a*).—Type A, Independent Tetrahedral Groups, $(SiO_4)^{-4}$. FIG. 4 (*b*).—Type B, Double Tetrahedra Structures, $(Si_2O_7)^{-6}$. FIG. 4 (*c*), (*d*), and (*e*).—Type C, Ring Structures.

(*c*) ring-linkage $(Si_3O_9)^{-6}$. (*d*) ring-linkage $(Si_4O_{12})^{-8}$. (*e*) ring-linkage $(Si_6O_{18})^{-12}$.

Another more widely used method of classification was based on the interpretation of silicates as salts of various hypothetical silicic acids. The most important of these are listed in Table IV. This method of classification is still used in most textbooks of mineralogy. It will undoubtedly be replaced by the modern classification according to crystal structure discussed below, but the change will require time.

Before we consider the names that

are in use in the classification according to crystal structure, it will be necessary to review briefly the most important features of the classification. The following treatment is taken with only minor changes from Berman (**1**) and Bragg (**2, 3**).

The classification of the silicates is based on the different types of silicon-oxygen arrangement which they display. The silicon-oxygen bond is the strongest in all the structures and the silicon-oxygen arrangement may be considered as the skeleton of a silicate structure. In many silicates the silicon may be partly replaced by aluminum. This alters the silicon-oxygen ratio, but such aluminum is reckoned with the silicon as part of the framework. The cations balance the valence requirements of the assemblage.

1. In all silicate structures examined, a silicon atom always occurs in the center of four oxygen atoms arranged as a tetrahedron about it. This tetrahedron is the fundamental and apparently invariable unit in the silicate structure. (Fig. 4 (*a*).)

2. The different silicate types arise from the various ways in which the silicon-oxygen tetrahedra in a given structure are related to one another. Tetrahedra can share corners only, and not sides or edges; consequently two tetrahedra can have only one oxygen in common between them. However, every oxygen of every tetrahedron may be shared with another tetrahedron.

3. Tetrahedra may be linked in only a few characteristic ways, as follows:[10]

(*a*) Independent tetrahedral groups: In this type, none of the tetrahedra shares corners with another. The resulting framework has the composition $(SiO_4)^{-4}$, (Fig. 4 (*a*)). Forsterite, Mg_2SiO_4, is of this type.

(*b*) Double tetrahedra structures: In this type, two tetrahedra share one oxygen, that is, are linked by one corner. The resulting framework has the composition $(Si_2O_7)^{-6}$ (Fig. 4 (*b*)). The melilite series $Ca_2Mg(Si_2O_7)$-$Ca_2Al(AlSiO_7)$ is of this type.

(*c*) Ring structures: In this type, two of the tetrahedral corners are shared, and the linkages are such as to form closed units of a ring-like structure. The resulting frameworks may have compositions such as $(Si_3O_9)^{-6}$, (Fig. 4 (*c*)), $(Si_4O_{12})^{-8}$ (Fig. 4 (*d*)), or $(Si_6O_{18})^{-12}$ (Fig. 4 (*e*)). Examples of this type are benitoite, $BaTiSi_3O_9$, and beryl, ideally $Be_3Al_2Si_6O_{18}$.

(*d*) Chain structures: In this type, two of the tetrahedral corners are shared, and the linkages are such as to form chains of indefinite extent. The resulting frameworks may be single chains of composition $(SiO_3)^{-2}$ (Fig. 3 (*a*)), or double chains of composition $(Si_4O_{11})^{-6}$ (Fig. 3 (*b*)). Examples of these types are the pyroxene acmite, $NaFe^{III}\,Si_2O_6$, and the amphibole tremolite, $Ca_2Mg_5Si_8O_{22}(OH)_2$.

(*e*) Sheet structures: In this type, three of the tetrahedral corners are shared, forming plane sheets of composition $(Si_2O_5)^{-2}$ (Fig. 2). Minerals of this type are characteristically platy; an example is talc, $Mg_3Si_4O_{10}(OH)_2$.

(*f*) Three-dimensional networks: In this type all four tetrahedral corners are shared, forming a continuous framework of composition SiO_2 (Fig. 1). All the forms of SiO_2 have this type of arrangement. If part of the silicon in the framework is replaced by aluminum, the valency is balanced by cations, as in orthoclase, $K(AlSi_3)O_8$, and analcime, $Na(AlSi_2)\,O_6\cdot H_2O$.

The foregoing brief outline gives the basis now used for silicate classification. It will be seen that when the framework

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[10] In the following descriptions, the framework has been given in terms of Si - O linkages, and the valences have been given as calculated on that basis. In all these, Si should be written as Z = largely Si, but with substitution by Al, and rarely by other elements. Such substitutions also change the valence balance.

TABLE V.—COMPARISON OF STRUCTURAL CLASSIFICATION WITH SILICIC ACID CLASSIFICATION.

Type of Structure	Framework Formula[b]	Corresponding Silicic Acid when Z = Entirely Si
A—independent tetrahedra	(ZO_4)	H_4SiO_4 — orthosilicic acid
B—double tetrahedra[a]	(Z_2O_7)	$H_6Si_2O_7$ — orthodisilicic acid
C—ring structures[a]	$(ZO_3)n$	H_2SiO_3 — metasilicic acid
D—chain structures	(ZO_3) (Z_4O_{11})	H_2SiO_3 — metasilicic acid
E—sheet structures	(Z_2O_5)	$H_2Si_2O_5$ — metadisilicic acid
F—three-dimensional networks	(ZO_2)	SiO_2

[a] Bragg (**2**) combined types B and C under the heading "Self-contained groups," that is, closed systems.

[b] Z is silicon chiefly, but may be substituted for in part by Al, rarely by other elements.

contains only silicon, there is good correspondence between the new classification and that based on the hypothetical silicic acids, as shown in Table V. However, when there is partial substitution in the framework, there is no correspondence between the two classifications. Thus, orthoclase and analcime above are in type F, but in the old classification are called a polysilicate and metasilicate, respectively.

The classification of the silicates by structural types suggested by Bragg (**2**) was accepted quite rapidly by mineralogists, but the names, as listed in Table V under "Type of Structure," were too awkward. Shorter names were needed. Table VI lists some of the names that have been used; most have been borrowed from the old classification by means of hypothetical silicic acids. It is not satisfactory to graft old names on to a new classification; confusion as to the meaning is an inevitable result, especially when the names are still in use in the old sense. It would seem wise, therefore, to abandon such terms as "disilicate" as designations for structural types. This has been done by Strunz, who has coined the new terms listed in the column *e* of Table VI. His names are unambiguous and much to be preferred over the others proposed. It will be noted that, following Bragg, he combines types B and C, both containing finite bounded groups. This combination is not strictly logical, as B and C have different types of linkages. It would therefore seem better to restrict the term sorosilicate to type B and to coin a new term for type C. I suggest the term cyclosilicate, using the Greek work for ring, which is in harmony with the Greek words used by Strunz.

CONCLUSION

I have tried to summarize present practices in the nomenclature of minerals and of some inorganic compounds, and to point out some of the important prob-

TABLE VI.—SOME OF THE NAMES SUGGESTED TO DESIGNATE TYPES OF SILICATE STRUCTURES.

Type of Structure	Framework Formula	Names Used to Designate Types*				
		a	*b*	*c* and *d*	*e*	*f*
A	(ZO_4)	Orthotype	Orthosilicate	Orthosilicate	Nesosilicates	Nesosilicates
B	(Z_2O_7)		Pyrosilicate	Orthodisilicate	Sorosilicates	Sorosilicates
C			Metasilicate	Metasilicate		Cyclosilicates
D	(ZO_3), (Z_4O_{11})	Metatype			Inosilicates	Inosilicates
E	(Z_2O_5)	Mica type	Disilicate	Metadisilicate	Phyllosilicates	Phyllosilicates
F	(ZO_2)	Feldspar type	Silica type	Dioxide type	Tektosilicates	Tektosilicates

* Column *a*—Machatschki (1932) (**14**).
Column *b*—Berman (1937) (**1**).
Column *c*—Swartz (1937) (**30**).
Column *d*—Winchell (1942) (**31**).
Column *e*—Strunz (1941) (**29**).
Column *f*—This paper.
Swartz reviews several other proposed classifications.

lems. It is apparent that there is much room for improvement. This can best be attained if those actively working in these fields give these matters careful consideration. Changes from established practices should be made only after thorough discussion and then, if possible, by agreement of widely representative groups. The individual scientist may use any nomenclature he pleases in his own notebook; he owes it to his fellows to discard his personal preferences, if necessary, and to conform to established notation in his published work. By so doing, he helps other scientists to understand what he writes, and that is the primary function of any nomenclature.

Acknowledgments:

Many of the thoughts expressed in this paper have been for a long time the subjects of discussions and friendly arguments with my colleagues of the Geological Survey. Many of the ideas herein were first suggested by them. I am also indebted to them for critically reading this paper and suggesting many improvements.

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DISCUSSION

Mr. L. A. Burkardt[1] (*presented in written form*).—The author of this paper has given an excellent survey of the origins and present state of nomenclature in mineralogy and inorganic chemistry. Fortunately, those interested in water-formed deposits face the problem of adapting existing nomenclature to their needs rather than contributing to such nomenclature.

Since most reports on the composition of water-formed deposits are made to persons with little or no chemical or mineralogical training, it would be advantageous if the nomenclature used afforded little opportunity for confusion of names or formulae. Names and formulae for more complex materials based on real or hypothetical acids or on crystal structure are more likely to be confusing than the use of trivial mineralogical names and the oxide type of formulae. In line with this mode of thinking, the calcium phosphates would be designated as whitlockite, $3CaO \cdot P_2O_5$, hydroxyapatite, $10CaO \cdot 3P_2O_5 \cdot H_2O$, and phosphorite, $3CaO \cdot P_2O_5 \cdot H_2O$; the use of the term hydroxyapatite being preferred to that of hydroxylapatite because of its more common use by the group at the present time.

With regard to the use of Greek letters and Roman numerals, it is suggested that the general practice of American mineralogists be followed. Where such usage is not clear, it is suggested that the Society's Committee D-19 on Water for Industrial Uses act to determine and publish the preference of the majority of the group interested in water-formed materials. It is further suggested that the nomenclature used by this group be considered at intervals by Committee D-19 and when variance with accepted usage of American mineralogists is noted, action be instigated by this committee to secure usage of nomenclature by this group which will conform to that of the mineralogists.

The use of a set of rules such as those suggested will secure a uniform method of reporting on the constitution of water-formed deposits, while occasional study and revision of the nomenclature used can serve to keep such nomenclature in conformity, as far as possible, with the general usage at that time among mineralogists.

Mr. T. H. Daugherty[2].—Mr. Fleischer mentioned that an ionic form of name or formula might be used for the more complex compounds in place of a formula based upon ratios of atomic equivalents or hypothetical oxides. While this suggestion appears to be desirable, I wonder whether sufficient data are available for the some fifty or fifty-five compounds pretty well known to occur in water-formed deposits in order to substantiate the use of ionic names or formulas?

Mr. Michael Fleischer (*author's closure*).—The paper, as printed, was restricted to the background of the problems involved, without going into the specific problems that you have. I think the rules suggested by your committee offer a good starting point. I would be inclined to change them slightly, as I shall mention in a moment, but the most important thing is for your group to adopt and adhere to one agreed set of rules. The next most important thing is to try to minimize the differences between your set and the mineralogists' set of rules.

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We are not going to get very excited about hydroxylapatite *versus* hydroxyapatite, or analcime *versus* analcite. The first of each pair is preferred by mineralogists, partly because of euphony (hydroxylapatite is easier to say), and partly because of priority. Mineralogy, like biology and botany, generally keeps the first namer's name, other things being equal.

There is a serious objection to the term phosphorite, as used by Mr. Burkardt for $Ca_3P_2O_8 \cdot H_2O$ (or $3CaO \cdot P_2O_5 \cdot H_2O$). The word phosphorite means to a geologist a rock composed largely of one or more calcium phosphates. It is therefore not the name of a definite mineral or chemical compound, and its use is likely to cause confusion. I suggest that $Ca_3P_2O_8 \cdot H_2O$ might better be called tricalcium phosphate monohydrate.

As I indicated, the condensed formula seems to me to be preferable to the oxide type of formula. Mr. Daugherty wonders whether we can write them that way, that is, do we have enough data to write them. I think we do for nearly all the compounds you have listed as occurring in boiler deposits. Take analcime, for example, I don't see why $NaAlSi_2O_6 \cdot H_2O$ is any harder to figure out than $Na_2O \cdot Al_2O_3 \cdot 4SiO_2 \cdot 2H_2O$ and I frankly see no advantage in the oxide type. Even for the most complicated one, such as cancrinite, you have to know as much about it to write the oxide formula as you do to write the condensed one. For cancrinite, these might be written in three different ways:

1. $Na_6Ca_2Al_6Si_6O_{24} \cdot 2CO_3 \cdot 3H_2O$
2. $6NaAlSiO_4 \cdot 2CaCO_3 \cdot 3H_2O$
3. $3Na_2O \cdot 2CaO \cdot 3Al_2O_3 \cdot 2CO_2 \cdot 6SiO_2 \cdot 3H_2O$

I do not see that the third of these is easier or more informative than the second, which has the advantage that it is much easier to indicate partial isomorphous substitution, say of K for Na. It should be remembered that all three formulas are only rough approximations, even for artificial material, and may be very far removed from the composition of the naturally occurring mineral cancrinite, which may contain potassium, sulfate, and chloride.

USE OF THE SPECTROSCOPE IN THE DETERMINATION OF THE CONSTITUENTS OF BOILER SCALE AND RELATED COMPOUNDS[1]*

By Alton Gabriel,[2] Howard W. Jaffe,[2] and Maurice J. Peterson[2]

Synopsis

The use of the simple bunsen spectroscope as a replacement for qualitative chemical tests in determining the metallic (cationic) constituents of boiler scales and related compounds apparently has not been emphasized. Over half the elements yield spectra in the visible range when subjected to arc excitation. Consequently the analyst has in the spectroscope an on-the-spot tool for examining samples and testing the purity of precipitates. He is thus able to vary and modify his analytical procedure to conform to the composition of the sample. For many routine uses within its field of usefulness, the spectroscope has the advantage of convenience over the spectrograph. The set-up of the instrument and its operation are described.

This talk stems from an impromptu demonstration of the utility of a small spectroscopic setup witnessed by A. A. Berk of A.S.T.M. Committee D-19 on Water for Industrial Uses. Since chemical spectroscopy has been known and practiced since the days of Kirchhoff and Bunsen during the middle of the past century and since our spectroscope has been used as a routine instrument for the past several years, it would not have voluntarily occurred to me that a talk on determinative spectroscopy would be a subject of current interest to this group.

Much has been written on the use of the spectrograph as an analytical tool. The paucity of recent literature on the utility of the simple spectroscope and the number of inquiries by visitors to our laboratory regarding its use, indicate that its usefulness has not been so well emphasized.

The several laboratories at the Eastern Experiment Station are frequently called upon to identify or to analyze quantitatively inorganic substances on which little or no information is furnished as to their chemical compositions or origins. Often a "complete analysis" is requested. Formerly, in order to determine the metallic constituents present in such samples, the analyst found it necessary (1) to follow through the entire scheme of qualitative chemical analysis, (2) to make individual tests chosen more or less at random, or (3) to make quantitative analyses for all suspected elements. Conditions 1 and 3 were found to be very time-consuming and 2 was frequently incomplete and entirely too subjective.

Uses of the Spectroscope

After due consideration of the problem, a spectrographic laboratory was established, and samples were examined spectrographically as an alternative to qualitative chemical analysis. The information so obtained was of inestimable value to the analysts for they were able to vary or to simplify procedures to conform with the composition of the samples to be analyzed. However, the spectrographic analyses were made on large spectrographs of high dispersion, which required a number of settings to cover the entire spectral range. The shifts to various portions of the spectrum required time-consuming adjustments in the instruments and of the accessory equipment on the optical benches. To minimize the needs for changing positions of the spectrographs, a small, 3-armed, bunsen-type spectroscope was set up in a location readily accessible to the analysts for use in determining the presence (or absence) of the alkalies. As the personnel became more proficient in the use of the instrument, its utility was found to be more extensive than was first anticipated. Soon it was being used for on-the-spot tests for determining the completeness of precipitations, for testing the purity of precipitates, and finally for detecting the cationic constituents in many of the samples themselves. As now operated, much of the qualitative work on the detection of the metallic constituents is performed on the spectroscope; the spectrograph being used for more formal requirements such as quantitative analyses, for the study of samples which yield spectra of unusual complexity, or in which a knowledge of the trace elements that may be present is of importance.

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[1] Published by permission of the Director, Bureau of Mines, U. S. Department of the Interior.

[2] Chemist-petrographer, Petrographer, and Spectrographer, respectively, Bureau of Mines, College Park Division, Metallurgical Branch, College Park, Md.

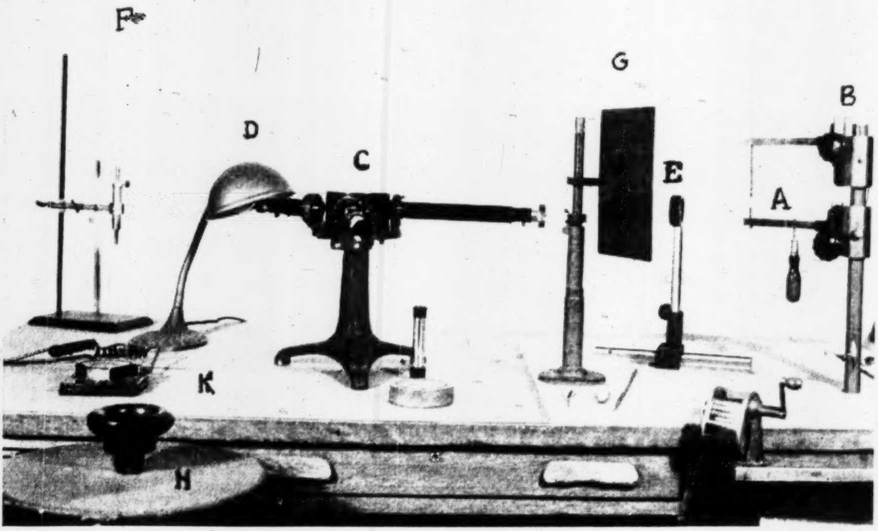

FIG. 1.—Spectroscopic Assembly.

A—Arc
B—Arc stand
C—Spectroscope
D—Light for illuminating the scale
E—Double convex lens
F—Electrode positioning screen
G—Metal shield to protect operator from direct radiation
H—Rheostat
I—Ammeter

There appears to be a more or less general belief that the spectroscope can be used for detecting only a relatively few elements—the alkalies and alkaline earths being mentioned most frequently. Actually over half of the elements in the periodic table yield spectra in the visible range. Most of these can be observed in a simple spectroscope if a power source sufficiently intense to cause excitation is used in conjunction with it. For example, if the simple bunsen flame is replaced by a d-c. arc supplied from a 220-v., 15-amp. line, the elements that can be detected if they occur as major or minor constituents include Li, Na, K, Ca, Sr, Ba, Ti, Zr, V, Cb, Cr, Mo, Mn, Fe, Co, Ni, Cu, Ag, Zn, Cd, Al, Si, Sn, Pb, and Bi, to mention only the partial list. In general, the anions, fluorine excepted, (**1**)[3] cannot be detected for they do not yield spectra in the visible range.

The use of the spectroscope in conjunction with an arc source for excitation is not new. The setup is used in the metallurgical industries for the rapid sorting of scrap metal and for control of production (**2**). One foreign manufacturer of optical equipment makes spectroscopes particularly designed for the determination of the constituents of ferrous and non-ferrous metals.

DESCRIPTION OF APPARATUS

The assembly used in our laboratory and its operation has been described by Peterson, Kauffman, and Jaffe (**3**). The apparatus is shown in Fig. 1. The light source *A* consists of a d-c. arc produced between two graphite electrodes. Power at 220 v. is supplied by a motor generator set. *H* is a variable rheostat used to control the power input to the arc and is capable of dissipating approximately 2500 w. The ammeter *K* has a total range of 25 amp. with subdivisions of $\frac{1}{2}$ amp.

The arc stand *B* consists of an upright bakelite post with two steel spring-clip arms attached in a horizontal position. These hold the graphite electrodes. Each electrode clamp is connected to a rack-and-pinion assembly mounted on the bakelite post and actuated by insulated knobs so that the electrodes can be vertically spaced by mechanical manipulation while the arc is burning. A permanently mounted clip spreader is used to spread the clip arms when changing the lower electrode. This tool is made from a screw driver by cutting off the steel portion approximately 2 in. above the handle. Two flat parallel surfaces are machined on the metal to allow the spreader to fit between the clips of the electrode clamp. Arc stands of various designs are offered for sale by manufacturers of spectrographic equipment.

The graphite electrodes used in this laboratory are $\frac{3}{16}$-in. diameter for the lower and $\frac{1}{8}$ or $\frac{3}{16}$-in. diameter for the upper. The lower electrode is cut to a length of $1\frac{1}{2}$ in. and one end is drilled approximately 7 mm. deep to provide a conical cavity for holding the sample. The upper electrode is pointed by means of the pencil sharpener. The "ordinary-grade" spectroscopic carbons are suitable for most chemical identification work, although traces of several elements present as impurities may be detected in them with the spectroscope.

The spectroscope, *C*, is a conventional 3-arm bunsen type constructed with a dispersing system consisting of two 60-deg. flint-glass prisms. The telescope arm can be swung through an arc to enable the observer to view any portion of the visible spectrum. By means of a hairline in the eyepiece, the exact positions of the spectral lines are determined relative to the illuminated scale which is imaged directly above the spectrum. A shielded incandescent light, *D*, is used to illuminate the scale.

A double convex lens, *E*, of approximately 5-in. focal length is used to project an image of the light source upon an electrode positioning screen, *F*, located about 4 ft. from the electrodes. The screen is marked to outline the exact position of the electrode image when the center of the arc and the slit are at the same vertical height. The correct vertical height and electrode gap are maintained during arcing by manipulation of the insulated knobs of the arc stand so that the electrode image is kept within the marked boundaries on the screen.

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[3] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1116.

TABLE I.—LOCATION OF CHARACTERISTIC GROUPS IN THE SPECTRA.

Element	Characteristic Groups[a]
Aluminum	A group of two lines (3961.5 and 3944.0) beyond the scale in the violet. Strong lines of Ca (3968.5 and 3933.7) form a group of four with the Al.
Calcium	A group of three lines in the green (5270.3—[5265.6—5264.2]—[5262.2—5261.7—5260.4]). Also a group of four lines in the blue (4456.6—[4455.9—4454.7]—4434.9—4425.4).
Calcium fluoride	A band head in the green (5291.0) graded toward the red.
Chromium	A group of three in the green ([5348.3—5345.8]—5328.4—5298.3).
Copper	A group of three lines in the green ([5220.1—5218.2]—5153.3—5105.6).
Iron	A group of five lines in the green (5371.5—5328.4—[5269.5—5266.6]—5232.9—5227.2).
Lithium	Two lines in the red (6707.9 and 6103.6).
Magnesium	A group of three lines in the green (5183.6—5172.9—5167.3).
Manganese	A group of five lines in the blue (4823.5—4783.4—4766.4—4762.4—4754.1).
Molybdenum	A group of three lines in the green (5570.5—5533.0—5506.5) also a group of three in the blue (4760.2—4731.5—4707.3).
Potassium	Two doublets—one off the scale in the red (7699.0 and 7664.9) and (6939.0 and 6911.3).
Sodium	The D lines in the yellow (5895.9 and 5889.9).
Titanium	A group of five lines in the green (5014.3—5007.0—4999.5—4991.1—4981.7).
Vanadium	A group of five lines in the blue (4881.5—4875.5—4864.8—4851.5—4807.6).
Zinc	A group of three lines in the blue (4810.5—4722.2—4680.1).

[a] Lines enclosed in brackets are considered as single lines, because in most cases they are not readily resolved.

A small hood, which is not shown in the photograph, is mounted above the arc stand to carry away the gases produced by the burning arc.

OPERATION OF THE SPECTROSCOPE

In actual operation, a 15- to 30-mg. sample is placed in the crater of a lower electrode and burned under a power input which is controlled by means of the rheostat. The behavior of the arc is essentially that of a furnace (**4, 5**) in which the low-boiling constituents, such as the alkalies, tend to volatilize first, followed by the intermediate elements, such as Sn, Cu, Fe, Ni, V, Cr, Si, Ba, and Ca, and finally the most refractory ones, including Al, Mo, Zr, Cb, W, etc. The progression of volatilization is not sharp, so overlapping between groups can be expected.

The current used and its control depend upon the composition of the sample. Materials, for example, with a high iron content are arced at 3 to 5 amp. to prevent sputtering and possible loss of the bead. The procedure with most samples is to operate the arc at 3 to 5 amp. for the first 2 min. then to raise the current to 10 to 12 amp. in order to volatilize the more refractory elements. When the more volatile constituents are suspected of being present or when it is desired to check the possible presence of an element occurring in relatively low concentration, the procedure is to set the telescope in a position to observe the lines of the particular element before striking the arc.

Direct-vision observation into the telescope tube of the spectroscope shows the emission from individual elements in the form of line spectra, each of which is a colored image of the slit. These lines are superimposed on a continuous spectral background, since no provision is made to prevent the light from the glowing electrodes from entering the slit of the spectroscope. The lines are identified in relation to an illuminated scale which is imaged directly above the spectrum. Band spectra, of course, also may appear superimposed upon the continuous background.

Scientifically trained personnel soon become proficient in handling the apparatus and in recognizing the spectrum of the respective elements. Many of the elements have their lines arranged in characteristic groups, the locations of some of which are given in Table I.

Many elements when present in major amounts can be quickly identified without a spectroscope through recognition of the colors they impart to the arc (6).

TABLE II.—CHARACTERISTICS THAT AID IN IDENTIFYING MAJOR CONSTITUENTS.

Element	Color on positioning screen or other characteristics
Al	Greenish blue
Sb	White fumes
As	Garlic odor
Ba	Green
Be	Greenish blue
B	Green
Ca	Orange
CaF_2	Canary yellow
Cb	Blue with red fringe—pitted lower electrode and white oxide coating
Cr	Green
Cs	Bluish white
Cu	Green
Fe	Blue with yellowish-white fringe—sparks and popping bead.
Li	Red
Mg	Green
Mo	Blue—metallic coating on lower electrode.
Nd	Light orange-red
K	Bluish-white
Pr	Greenish-gray
Sc	Light orange
Sm	Red
Ag	Green
Na	Yellow
Sr	Red
Ta	Blue with a red fringe—pitting on lower electrode and white oxide coating
Tl	Green (very intense)
Ti	White
U	Bluish white
W	Blue when current is reduced—pitting of lower electrode and yellow oxide coating.
Y	Red
Zr	White flashes

They will be imaged on the test screen in the form of a halo surrounding the center portion of the arc.

The characteristics shown in Table II will aid in identifying major constituents.

RESULTS OF TESTS ON BOILER SCALE MATERIALS

Through the courtesy and cooperation of certain laboratories, a number of samples of boiler scale and allied materials were made available to the authors for spectroscopic examination. The results of the study are summarized as follows:

In the examination of 33 samples on which chemical analyses were furnished, no trouble was experienced in determining the major and minor cationic constituents. The elements encountered included Na, Ca, Al, Si, Fe, Cu, Mg, Zn, Ti, Mn, Ba, Li, V, Pb, Ni, Sn, and F.

In several instances a spectroscopic study of the sample preliminary to its quantitative chemical analyses would have been advantageous. In one group of analyses, soda, zinc, copper, magnesium, and aluminum were reported as being negative, presumably after quantitative analyses had been made for them. A spectroscopic examination would have revealed the presence (or absence) of these elements at a moment's glance. It would have saved the analyst the time spent in obtaining negative results.

TABLE III.—REPORT ON EXAMINATION OF SAMPLES BY SPECTROSCOPIST AND CHEMIST.

Elements Detected By		Sample No. 1		Sample No. 2		Sample No. 3	
Spectroscopist	Chemist	Spectroscopist[a]	Chemist	Spectroscopist	Chemist	Spectroscopist	Chemist
Na		vs		vs			
V	V_2O_5	s	5.4	vs	20.5	m	5.3
Fe	Fe_2O_3	vs	30.9	s	14.6	vs	42.0
Ni	NiO	m	2.9	s	5.0	n.d.	0.4
Mg	MgO	m	0.1	m	0.1	n.d.	0.2
Si	SiO_2	n.d.	0.6	n.d.	1.2	m	0.6

[a] Spectroscopist's report: vs = very strong; s = strong; m = minor; n.d. = not detected.

In several instances constituents were not determined chemically although they occurred in minor and even in major amounts. Zinc, in particular, was found to have been frequently overlooked. One sample carefully analyzed for SiO_2, Fe_2O_3, P_2O_5, CaO, MgO, Al_2O_3, CO_2, and SO_3 was found by spectroscopic examination to contain zinc in more than minor amounts, also copper and some fluorine.

Subsequent chemical analyses revealed the presence of 2.47 per cent zinc, 0.92 per cent copper, and 0.38 per cent fluorine.

The utility of the spectroscope in the examination of samples preliminary to chemical analysis is illustrated in the case of three air preheater deposits. The spectroscopist's report on the constituents and the subsequent analyses are shown in Table III.

In several instances discrepancies between furnished analyses and spectroscopic findings occurred. These have been brought to the attention of the respective laboratories. It is hoped that comments concerning them will be made by company representatives at the round table discussion.

Many laboratories are unable financially to equip and support a modern spectrographic unit. A small spectroscopic setup such as that illustrated in Fig. 1 represents only a modest investment. If intelligently used, such a setup should be a valuable adjunct to the average analytical or petrographic laboratory.

Acknowledgments:

The investigation was made under the general supervision of J. B. Zadra, Chief, College Park Division, Metallurgical Branch. The cooperation of the following companies in furnishing samples is gratefully acknowledged: W. H. and L. D. Betz, Combustion Engineering Co., Inc., Allis-Chalmers Manufacturing Co., The Dow Chemical Co., and Hall Laboratories, Inc. Appreciation is expressed to A. A. Berk for suggesting the paper.

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DISCUSSION

Mr. Frank E. Clarke.[1]—I concur with the authors that without spectroscopic aid, analysis of such a complex material as boiler scale is a very difficult problem. The fact that an analysis totals 100 per cent ± 1 or 2 per cent does not mean very much, particularly if it is reported in terms of probable constituents, since the values may be based on erroneous reasoning.

The zinc problem mentioned is a common one. On one occasion we lost 12 per cent of zinc in the analysis of a boiler deposit, and without reliable spectrographic data it would have been difficult to determine the constituent responsible for the low total. Zinc is not the only constituent which escapes detection in the analysis of boiler deposits. Lead also is elusive and compounds which precipitate in perchloric acid (for example, barium sulfate) frequently are reported as silica unless the residue is treated with hydrofluoric acid. Titanium also precipitates with silica under certain conditions, and vanadium frequently is precipitated and reported as R_2O_3. One can think of many similar examples.

The Station never analyzes a boiler deposit without first making a semiquantitative spectrographic evaluation. We use a grating spectrograph rather than a spectroscope in this work and find it to be an invaluable aid. It is my personal opinion that it would be very difficult to make rapid, accurate analyses of complex boiler deposits without the aid of spectroscopic or spectrographic analysis.

I was particularly interested in this paper because the spectroscope is a relatively simple and inexpensive device which should be available to any laboratory. The Station's spectrograph with its accessories cost approximately $10,000. It should also be noted that except for the direct reading spectrograph, which is very much more expensive, the spectroscope is a more rapid means of qualitative evaluation.

Mr. R. K. Scott[2].—We have been doing some work with the spectrograph recently, and are being continually surprised at its usefulness. The chief drawback to the spectrograph, however, is the high initial cost of equipment suitable for analysis of all types of water-formed deposits encountered in the commercial laboratory. The authors have presented a method which not only puts the initial cost within the range of a great many laboratories, but one which becomes even more rapid than the photographic method for qualitative analysis.

In at least one of the six samples which we submitted to the authors, spectroscopic examination prior to chemical analysis would have been of very great help to the chemist. We had found titanium qualitatively in this sample and at first believed it to be a major constituent, since much of the sample remained as a "silica" residue. Including the time spent in separating the residue and determining that it was largely iron instead of titanium and rerunning the analysis, the chemist spent considerable additional time to accomplish what the spectroscope could have determined in a few minutes. Minor constituents, whose determination had been omitted in the chemical analyses, were detected by the spectroscope in five of the six samples. Additional information which would be of potential use if obtained regularly on all samples was reported by the spectroscopist in every sample.

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[1] Senior Chemist, Head of Physical and Chemical Branch, Chemical Laboratory, U. S. Naval Engineering Experiment Station, Annapolis, Md.

[2] Hall Laboratories, Inc., Pittsburgh, Pa.

TABLE IV.—COMPARISON OF CHEMICAL AND SPECIAL ANALYSES.

Sample	Chemical Analysis, per cent	X-Ray Analysis	Arc Spectroscope Analysis		
			Above 5 per cent	0.5 to 5 per cent	Less than 0.5 per cent
Boiler scale no. 1	SiO_2 36.6 R_2O_3 31.9 CaO 10.4 P_2O_5 2.7 SO_3 0.6 Ign. 2.9 Cu 2.3 MgO 0.4	Acmite $Na_2O \cdot Fe_2O_3 \cdot 4SiO_2$ Hydroxyapatite $10\ CaO \cdot 3P_2O_5 \cdot H_2O$ Copper Cu	Sodium Copper Iron Calcium Silicon	Magnesium Zinc Nickel Aluminum Chromium	Manganese
Boiler scale no. 2	SiO_2 5.1 R_2O_3 18.3 CaO 21.7 MgO 8.2 P_2O_5 2.8 CO_2 16.9 Ign. 22.3 Cu 18.0		Copper Magnesium Calcium Iron	Aluminum Manganese	Fluorine Sodium Titanium Zinc Nickel Silicon
Turbine deposit (corrosion)	...	Chalcocite Cu_2S	Copper	Zinc Tin Nickel Silicon	Lead Iron
Condenser deposit (corrosion)	...	$ZnCO_3$ ZnO	Zinc Copper		

An additional advantage has been mentioned by the authors, in that some elements which are ordinarily determined are not found in significant amounts in certain samples, indicating that their determination could be omitted in the course of running the chemical analysis. This would result in a considerable saving of time. The spectroscope will indicate very readily whether any of the heavy metals which require sulfide separation are present in significant amounts.

The inability of the spectroscope to detect most anions is not serious, as rapid qualitative tests are available for practically all of these. The chief difficulty in the use of the spectroscope on water-formed deposits is the determination of minor amounts of silica, since the only strong line occurs almost in the ultraviolet, where the sensitivity of the eye is very low.

I think the authors have done a beautiful job of presenting a really workable method for qualitative analysis of the cationic constituents of water-formed deposits.

MR. MICHAEL FLEISCHER.[3]—In view of what has been said, perhaps another testimonial is unnecessary, but our work, which consists of examining samples of a very wide range in type, is such that we had a rule for a long time that complete analyses are not made prior to spectrographic analysis. We have two very expensive spectrographs.

Unfortunately, however, we have had only one spectrographer for some time and the result is that many times we do not wait for him. We have to do the analyses, and there are many stories of what happens then. I undertook an analysis and kept getting queer precipitates, R_2O_3 that fused when ignited, and all sorts of weird, wonderful things, and finally it turned out that the sample had 1½ per cent of V_2O_5 in it which precipitated over everything. It came down with the copper sulfide, and it came down with the iron, and it came down everywhere else. The result was that three weeks were wasted. That could have been avoided by a spectrographic analysis.

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[3] Senior Geochemist, U. S. Department of the Interior, Geological Survey, Washington, D. C.

The best testimonial to the instrument that I can give you is to say that a few months ago we went out to College Park and looked at it, and we bought one.

MR. C. E. IMHOFF.[4]—The samples sent by our laboratory to Messrs. Gabriel, Jaffe, and Peterson for examination under the arc spectroscope were those on which no attempt had been made to determine quantitatively the minor constituents. In a routine chemical analysis, these minor metallic constituents are usually precipitated by ammonium hydroxide and are all weighed together as what is termed R_2O_3. The accompanying Table IV indicates what information the arc spectroscope reveals in addition to chemical analysis and X-ray analysis. Considering the spectroscopic analysis of scale No. 1, the constituents appearing in greater amounts than 5 per cent are all confirmed by chemical or X-ray analysis except for copper. Copper is definitely present and a referee analysis would be necessary in order to determine how close to 5 per cent the actual copper concentration was in the sample. Zinc, nickel, aluminum, chromium, and manganese are all hidden in the R_2O_3. Usually these constituents are not too important in minor amounts in a boiler scale, although they may be important indicators if they exist in corrosion deposits.

The spectroscopic analysis of scale No. 2 is in agreement with the major constituents of the chemical analysis in all respects except for silica. The spectroscopic analysis indicates this to be less than 0.5 per cent. Spectrographers have had difficulty in the quantitative determination of silicon and it is apparent that the combination of associated materials in this scale are not favorable for the quantitative detection of silica in this instance. Fluorine, one of the minor constituents, is indicated and it is extremely interesting to know that fluorine can be determined spectroscopically. The determination of fluorine chemically is not too easy and the procedure is quite lengthy.

In regard to the turbine corrosion deposit, X-ray analysis indicated nothing but chalcocite, whereas the spectroscope showed that there were considerably more elements present as minor impurities in not sufficiently large amounts to give any diffraction pattern. In the condenser deposit, the X-ray revealed only zinc carbonate and zinc oxide, whereas the arc spectroscope showed the presence of copper in addition to that of zinc.

From an observation of these comparisons and the description of the use of the arc spectroscope, it is apparent that it can be a very useful tool in the examination of scales and deposits, especially when used in conjunction with chemical analysis and some of the other special methods of examination. If a particular constituent is present in amounts great enough to be considered quantitatively, a chemist will take steps towards its isolation and determination, otherwise, it can be ignored and determined in group precipitation such as R_2O_3.

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[4] Water Conditioning Dept., Allis-Chalmers Mfg. Co., Milwaukee, Wis.

MR. ALTON GABRIEL (*author's closure by letter*).—During the course of this investigation, visual spectroscopic analyses were made on a number of samples of boiler scale and related compounds submitted by various commercial laboratories. The constituents found in the respective samples were classified as major constituents (above 5 per cent), minor constituents (0.5 to 5 per cent), and tract constituents (less than 0.5 per cent). These percentage limits were chosen more or less arbitrarily but were found to be satisfactory for samples of complex composition.

While the detection of a particular element is extremely objective, the estimation of its concentration in a sample is to a large extent subjective. In brief, the observation of certain characteristic spectral lines establishes the identity of the element in question. Its concentration, however, is estimated from the intensity of the lines and is subject to several variables. These include the experience of the operator in judging intensities, possible interference by lines of other elements, characteristics of the particular instrument used and the variance in the base material of different samples. Consequently values given for concentration are estimates in the true sense of the word, and while they are not quantitative, they are more than "idle shots in the dark."

In cases when spectroscopic estimates could be checked with chemical analyses, the correlation was surprisingly good. Spectroscopic estimates for silica were low in one or two cases. This may be expected, since the only line of silicon in the visible spectrum lies in the near ultraviolet and is relatively insensitive to the eye. In a number of instances, however, constituents were found spectrographically that had been omitted in the chemical analyses.

Indications are that the analyst could save considerable time, expense, and avoid many difficulties by adopting a policy of having unknown samples examined spectroscopically prior to chemical analysis. As a further safeguard against problematical determinations he may also examine his precipitates spectroscopically for purity. To quote Mr. R. H. Scott of Hall Laboratories, "Additional information which would be of potential use if obtained regularly on all samples was reported by the spectroscopist in every sample."

AN EVALUATION OF TEST METHODS FOR THE DETERMINATION OF DISSOLVED OXYGEN IN DEAERATED BOILER FEEDWATER*†

By J. F. Sebald[1]

The need for a standardized dissolved oxygen test method as a means for defining dissolved oxygen performance for deaerating equipment was first recognized by the Heat Exchange Institute during the latter part of 1942. Performance levels had been established for some years, but the manner in which performance was to be measured was left to the discretion of the deaerating equipment manufacturer, the consumer, or the consumer's engineering representative.

In many instances the lack of specific knowledge in the field of dissolved oxygen testing left the consumer in doubt as to the actual performance of his deaerating machinery.

Much has been contributed in the field of dissolved oxygen determination in boiler feedwaters, but little correlation of this information has been undertaken. In the absence of such a correlation of data with respect to type of test and the test procedures from the standpoint of test accuracy and precision, wide differences in opinion on test validity exist. In the early stages of the work conducted by the Heat Exchange Institute, one of the member companies was given the assignment to propose a test method and present it in complete form for comment by other member companies of the Institute.

The results of this survey among the members of the Heat Exchange Institute showed an almost complete lack of agreement as to test method and little agreement as to test procedure. This lack of agreement on the part of successful manufacturers of deaerating equipment was in one sense encouraging. Since they represented a group who were particularly familiar with dissolved oxygen testing, their diversity of opinion quite logically indicated that the differences in the accuracy and precision of the test methods and procedures generally accepted by the trade were not beyond reconciliation. While disagreement as to methods and procedures existed at this time, the survey showed more clearly the need for selecting a single method and reliable procedure to be endorsed by the Heat Exchange Institute, and initiated the cooperative effort of the member companies toward this end.

The Heat Exchange Institute Oxygen Test Methods Committee was organized in 1943. Its assignment was to select a single test method worthy of endorsement and to develop a test procedure which could be relied upon to achieve a high order of test precision. The first meeting of this committee was held in 1944 and the area for investigation was established.

SELECTION OF TEST METHODS FOR EVALUATION

Four test methods generally accepted as reliable were considered for evaluation as to performance: the Winkler (**1**)[2], the Schwartz-Gurney "A," (**2**) the Schwartz-Gurney "B," (**3**) and the U. S. Navy Laboratory modification of the Schwartz-Gurney "A" (**4**).

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* Presented at the Fiftieth Annual Meeting of the Society, June 16–20, 1947.

† The material included herein has been developed under the sponsorship of the Heat Exchange Inst.

[1] Chairman, Oxygen Test Methods Committee, Heat Exchange Institute, and Assistant Chief Engineer, Condenser and Heater Engineering Div., Worthington Pump and Machinery Corp., Harrison, N. J.

Winkler Test:

The Winkler test was considered because of its simplicity, and, although it does not correct for interfering compounds in waters, it may readily be used for boiler feedwaters of high purity. It has the disadvantage of requiring a correction for dissolved oxygen added with the fixing reagents and requires absolute accuracy with respect to the normality of the titrating reagents. Because most boiler feedwaters contain some interfering compounds in solution, the use of the Winkler test may be very limited.

Schwartz-Gurney "A" Test:

The Schwartz-Gurney "A" (**2**) test was considered because it is the most easily conducted of the interference compensating methods. Although it has not enjoyed much popularity since the advent of the Schwartz-Gurney "B" method, it was used by the U. S. Navy Laboratory for acceptance tests for naval deaerators until 1942. Experience with the test indicated that it had too much to offer to be excluded from the evaluation. It has the disadvantage of requiring a correction for the dissolved oxygen added with the reagents.

Schwartz-Gurney "B" Test:

The Schwartz-Gurney "B" (**3**) test was considered because of its general popularity as an interference compensating test which, in addition, corrects for the dissolved oxygen added with the reagents as well as for reagent impurity. It is complicated because of the three samples required for each test and it requires double-size samples to establish the same degree of precision as the other test methods selected. The use of double-size sample-collection flasks to compensate for this inherent limitation is awkward.

U. S. Navy Laboratory Method:

The U. S. Navy Laboratory modification of the Schwartz-Gurney "A" test was considered because of its generally accepted chemical superiority. The advantage in producing iodine reactions in the presence of free iodine is well known in the field of idiometric measurement (**5**), and much credit is due Adams, Barnett, and Keller (**4**) for applying this refinement to the Schwartz-Gurney "A" test. The test has the disadvantage of occasionally requiring a change in the strength of the free iodine-containing reagent until the interference and dissolved oxygen levels are established. It also requires a correction for the dissolved oxygen added with the reagents.

AREA OF INVESTIGATION

For establishing the area to be investigated, consideration was given to the standard dissolved oxygen performance requirements as recommended by the Heat Exchange Institute to meet the trade conditions. These requirements, usually specified as 0.005 or 0.03 cu. cm. per liter, established the oxygen levels at which the tests were to be conducted. For well deaerated boiler feedwaters it was desirable to conduct tests with oxygen levels approaching zero. For less well deaerated boiler feedwaters, the level selected for investigation was between 0.015 and 0.03 cu. cm. per liter of dissolved oxygen.

In addition to performance at the two oxygen levels selected, further investigation of performance was to be made at the same oxygen levels in presence of known amounts of interference. The interfering compounds selected were ferrous iron, ferric iron, tannic acid, and a mixture of all three. These compounds were considered as the most likely to be present in boiler feedwater and of the type which offered the most likelihood for complete correction.

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[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1137.

The test method which seemed to offer the most in accuracy and in precision was to be subjected to further test using sodium sulfite as the interfering compound. This preferred test method was also to be used for the determination of the magnitude of reagent correction as well as for the evaluation of the precision of starch titration.

SELECTION OF ANALYTICAL EQUIPMENT AND STIPULATION OF TEST PROCEDURE

The selection of glassware (Fig. 1) by the committee was made on the basis of reliability and convenience. The McLean flask selected varied somewhat from that recommended by the A.S.T.M.[3] in that the end closures were made conical instead of hemispherical to eliminate horizontal surfaces to which air bubbles have a tendency to collect and are difficult to dislodge. The flask extension tubes have no enlargement to reduce length, as it was considered difficult to wash the A.S.T.M.

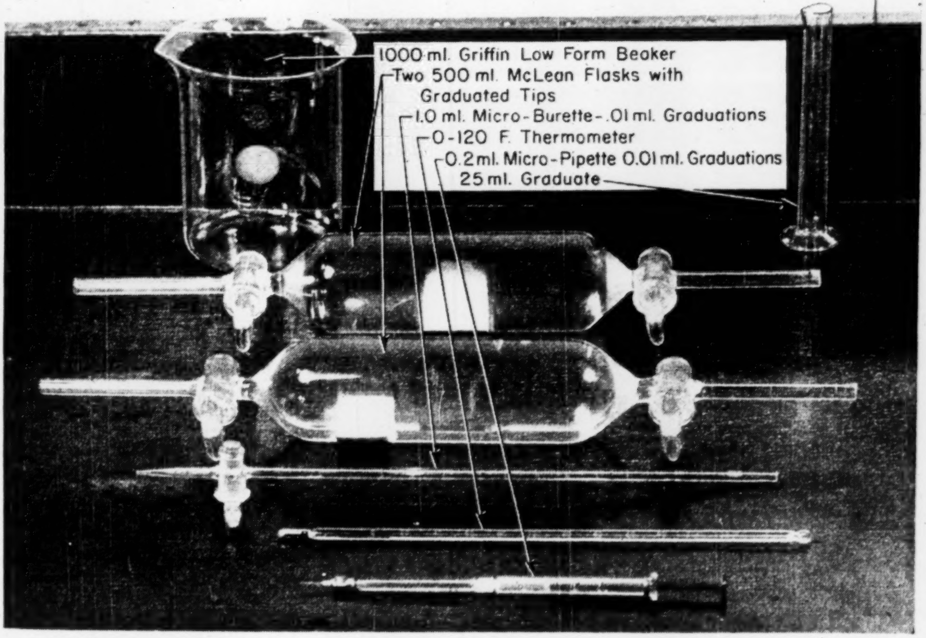
1000 ml. Griffin Low Form Beaker
Two 500 ml. McLean Flasks with Graduated Tips
1.0 ml. Micro-Burette-.01 ml. Graduations
0-120 F. Thermometer
0.2 ml. Micro-Pipette 0.01 ml. Graduations
25 ml. Graduate

FIG. 1.—Glassware Used in Oxygen Test Methods Committee Test Procedure.

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[3] Tentative Methods of Test for Dissolved Oxygen in Industrial Water (D 888 – 46 T), 1946 Book of A.S.T.M. Standards, Part III-A, p. 1020.

type clean between the addition of reagents. For the addition of sodium thiosulfate for the titration, a 1-cu. cm. microburette graduated in 0.01-cu. cm. divisions was used for the initial phase of the titration with the final end point titration made with a pipette graduated in 0.01-cu. cm. divisions with mechanical control similar to titrating apparatus used in microanalysis.

The test procedure used, as well as the apparatus, was in substantial agreement with that described by the author in 1944 (6) and no changes in apparatus or procedure were found necessary throughout the investigation.

TABLE I.—PASSAIC VALLEY WATER COMMISSION, RESULTS OF CHEMICAL ANALYSES—FILTRATION WORKS, LITTLE FALLS, N. J.

Parts per million

1946 mixed supply; filtered Wanaque plus filtered Passaic

	Temperature, deg. Fahr.	Dissolved oxygen	Free ammonia	Alb. ammonia	Nitrites	Nitrates	Hardness	Alkalinity	Incrustants	Total residue	Iron	Chlorides	Carbon dioxide	Sulfates	Sodium	Magnesium	Calcium	Aluminum oxide	pH	Silica	Manganese
1946 yearly average	54	9.5	0.095	0.101	0.00	0.24	51	31	20	87	0.05	3.9	2	23.5	4.7	4.2	15.0	1.0	7.3	3.3	0.04
1946 maximum	70	13	0.168	0.170	0.002	0.46	66	41	28	109	0.07	6.5	3	27	7.2	5.6	17	1.3	7.6	4.1	0.09
1946 minimum	35	6.8	0.034	0.016	0	0.16	38	21	12	59	0.00	2.0	1	21	3.2	3.5	12	0.6	7.0	2.3	0.03

All titrations were conducted by potentiometric means, using calomel and platinum electrodes. The instrument used for measuring the voltage was a vacuum tube voltmeter of the direct reading type.

Starch titrations were made for comparing the precision of the interference compensating test when conducted with potentiometric and starch titrations.

SELECTION OF TEST SITE

In selecting the test site, the committee considered the type of water available, location for the convenience of supervision, plant facilities, and available personnel. The site selected had three sources of water, namely, condensate, spray pond water, and city water, any of which showed promise of being satisfactory. After some preliminary test runs and inspections of water analyses, the city water supply was chosen as the best available medium for conducting the experiments. Table I shows the average analysis of the water and the maximum variations for the year of 1946, during the time the investigation was made. Occasional water analyses made at the test site showed close agreement with the results shown in Table I, provided through the courtesy of Passaic Valley Water Commission, Filtration Works, Little Falls, N. J.

SELECTION OF MECHANICAL EQUIPMENT

In an effort to establish test data which could not be considered as specifically applying to laboratory conditions, the deaerator selected was identical with that used for the mobile power trains built during the recent war. Modifications were made to this deaerator by adding a second stage of deaeration in series with the first stage (Fig. 2) in order to effect extreme deaeration in the effluent. The equipment (Fig. 3) consisted of a deaerator complete with water inlet control and measuring device, water outlet control, steam regulator, chemical feed pump of the constant-speed variable-stroke type, chemical mixing tank, multiple orifice type chemical mixer in water supply

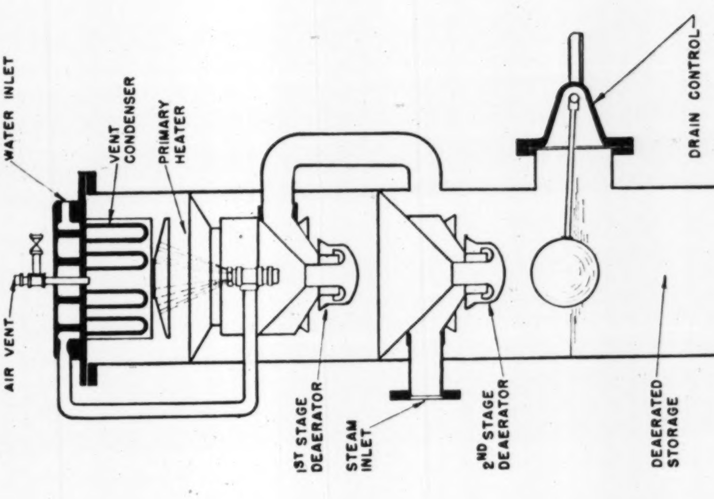
AIR VENT
WATER INLET
VENT CONDENSER
PRIMARY HEATER
1ST STAGE DEAERATOR
STEAM INLET
2ND STAGE DEAERATOR
DEAERATED STORAGE
DRAIN CONTROL

FIG. 2.—Schematic Drawing of Deaerator Equipment.

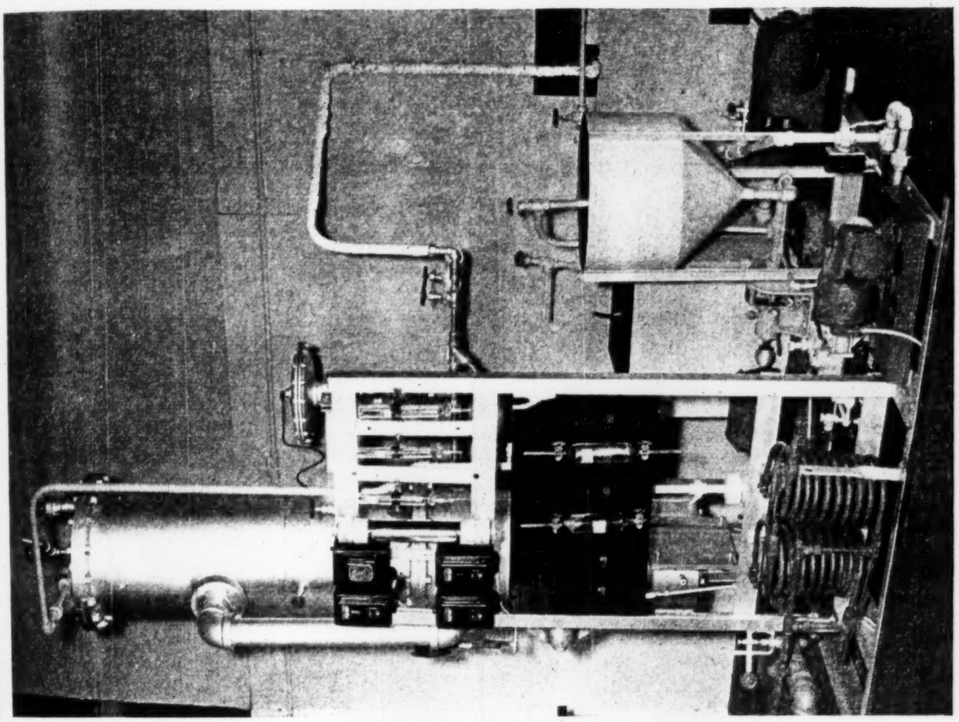

FIG. 3.—Deaerator and Sampling Station.

line, water supply reservoir, and transfer pump. All equipment used other than the measuring instruments was of commercial type and size. This mechanical equipment was housed in a temporary room 18 ft. square, erected in a large shop building. The analytical station (Fig. 4) was housed in the same room for convenience and was somewhat more elaborate than that usually found in the field, justifiable, however, on the basis of the long duration of the test.

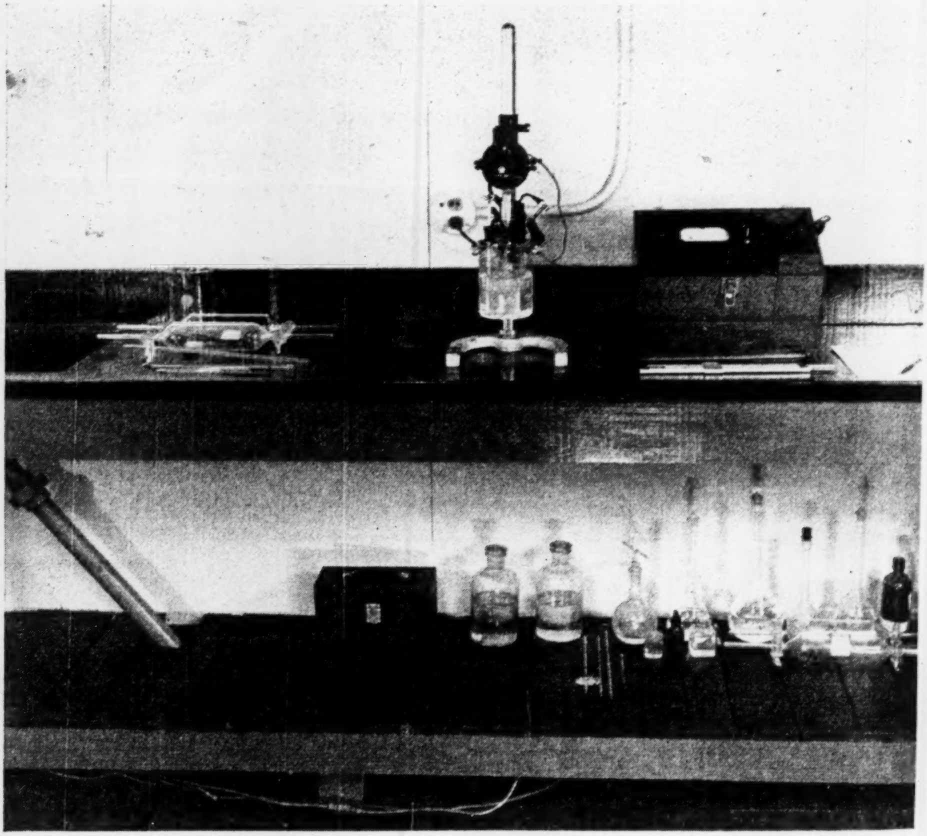

FIG. 4.—Analytical Station Used in the Test Procedure.

THE TEST AGENDA

The test program was divided into two parts. The first part was for the evaluation of accuracy and precision for the purpose of selecting a preferred test method; the second part was for the purpose of further evaluation of the preferred method and includes reagent-correction tests and tests for the determination of the precision of starch titration.

The methods considered—the Winkler, the Schwartz-Gurney "A," the Schwartz-Gurney "B," and the U. S. Navy Laboratory modification of the Schwartz-Gurney "A"—were conducted with potentiometric titration.

PART I OF TEST PROGRAM

Test No. 1:

1. Water Conditions
   - (*a*) city supply,
   - (*b*) interference—negligible, and
   - (*c*) oxygen content—0.0000 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
   - (*a*) Winkler
   - (*b*) Schwartz-Gurney "A"
   - (*c*) Schwartz-Gurney "B"
   - (*d*) U. S. Navy Laboratory method
3. Number of Tests
   - (*a*) 20 to 40 for each method.
4. Purpose of Tests
   - (*a*) precision in absence of dissolved oxygen
   - (*b*) accuracy in absence of dissolved oxygen.

Test No. 2:

1. Water Conditions
   - (*a*) city supply,
   - (*b*) interference—negligible, and
   - (*c*) oxygen content—0.015 to 0.03 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
   - (*a*) Winkler,
   - (*b*) Schwartz-Gurney "A,"
   - (*c*) Schwartz-Gurney "B," and
   - (*d*) U. S. Navy Laboratory method.
3. Number of Tests
   - (*a*) 5 to 10 for each method.
4. Purpose of Tests
   - (*a*) Accuracy in absence of dissolved oxygen.

Test No. 3:

1. Water Conditions
   - (*a*) city supply,
   - (*b*) interference—1 to 2 ppm. ferric sulfate, and
   - (*c*) oxygen content—0.0000 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
   - (*a*) Winkler,
   - (*b*) Schwartz-Gurney "A,"
   - (*c*) Schwartz-Gurney "B," and
   - (*d*) U. S. Navy Laboratory method.
3. Number of Tests
   - (*a*) 5 to 10 for each method.
4. Purpose of Tests
   - (*a*) effect of interference—dissolved oxygen absent.

Test No. 4:

1. Water Conditions
   - (*a*) city supply,
   - (*b*) interference—1 to 2 ppm. ferrous sulfate, and
   - (*c*) oxygen content—0.0000 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
   - (*a*) Winkler,
   - (*b*) Schwartz-Gurney "A,"
   - (*c*) Schwartz-Gurney "B," and
   - (*d*) U. S. Navy Laboratory method.
3. Number of Tests
   - (*a*) 5 to 10 for each method.
4. Purpose of Tests
   - (*a*) effect of interference—dissolved oxygen absent.

Test No. 5:

1. Water Conditions
   - (*a*) city supply,
   - (*b*) interference—1 to 2 ppm. tannic acid, and
   - (*c*) oxygen content—0.0000 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
   - (*a*) Winkler,
   - (*b*) Schwartz-Gurney "A,"
   - (*c*) Schwartz-Gurney "B," and
   - (*d*) U. S. Navy Laboratory method.
3. Number of Tests
   - (*a*) 5 to 10 for each method.
4. Purpose of Tests
   - (*a*) effect of interference—dissolved oxygen absent.

Test No. 6:

1. Water Conditions
   - (*a*) city supply,
   - (*b*) interference—1 to 2 ppm. ferric and ferrous sulfates and tannic acid, and
   - (*c*) oxygen content—0.0000 cu. cm. per liter.

2. Test Methods Employed—Potentiometric Titration
   - (a) Winkler,
   - (b) Schwartz-Gurney "A,"
   - (c) Schwartz-Gurney "B," and
   - (d) U. S. Navy Laboratory method.
3. Number of Tests
   - (a) 5 to 10 for each method.
4. Purpose of Tests
   - (a) effect of interference—dissolved oxygen absent.

Test No. 7:

1. Water Conditions
   - (a) city supply,
   - (b) interference—1 to 2 ppm. ferric sulfate, and
   - (c) oxygen content—0.015 to 0.03 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
   - (a) Winkler,
   - (b) Schwartz-Gurney "A,"
   - (c) Schwartz-Gurney "B," and
   - (d) U. S. Navy Laboratory method.
3. Number of Tests
   - (a) 5 to 10 for each method.
4. Purpose of Tests
   - (a) effect of interference—dissolved oxygen present.

Test No. 8:

1. Water Conditions
   - (a) city supply,
   - (b) interference—1 to 2 ppm. ferrous sulfate, and
   - (c) oxygen content—0.015 to 0.03 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
   - (a) Winkler,
   - (b) Schwartz-Gurney "A,"
   - (c) Schwartz-Gurney "B," and
   - (d) U. S. Navy Laboratory method.
3. Number of Tests
   - (a) 5 to 10 for each method.
4. Purpose of Tests
   - (a) effect of interference—dissolved oxygen present.

Test No. 9:

1. Water Conditions
   - (a) city supply,
   - (b) interference—1 to 2 ppm. tannic acid, and
   - (c) oxygen content—0.015 to 0.03 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
   - (a) Winkler,
   - (b) Schwartz-Gurney "A,"
   - (c) Schwartz-Gurney "B," and
   - (d) U. S. Navy Laboratory method.
3. Number of Tests
   - (a) 5 to 10 for each method.
4. Purpose of Tests
   - (a) effect of interference—dissolved oxygen present.

Test No. 10:

1. Water Conditions
   - (a) city supply,
   - (b) interference—1 to 2 ppm. ferric and ferrous sulfates and tannic acid, and
   - (c) oxygen content—0.015 to 0.03 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
   - (a) Winkler,
   - (b) Schwartz-Gurney "A,"
   - (c) Schwartz-Gurney "B," and
   - (d) U. S. Navy Laboratory method.
3. Number of Tests
   - (a) 5 to 10 for each method.
4. Purpose of Tests
   - (a) effect of interference—dissolved oxygen present.

PART II OF TEST PROGRAM

Test No. 11:

1. Water Conditions
   - (a) city supply,
   - (b) interference—14 ppm. sodium sulfite, and
   - (c) oxygen content—0.0000 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
   - (a) preferred method.
3. Number of Tests
   - (a) 5 to 10 for each method.
4. Purpose of Tests
   - (a) effect of interference—dissolved oxygen absent.

Test No. 12:

1. Water Conditions
    - (*a*) city supply,
    - (*b*) interference—28 ppm. sodium sulfite, and
    - (*c*) oxygen content—0.0000 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
    - (*a*) preferred method.
3. Number of Tests
    - (*a*) 5 to 10 for each method.
4. Purpose of Tests
    - (*a*) effect of interference—dissolved oxygen absent.

Test No. 13:

1. Water Conditions
    - (*a*) city supply,
    - (*b*) interference—14 ppm. sodium sulfite, and
    - (*c*) oxygen content—0.015 to 0.03 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
    - (*a*) preferred method.
3. Number of Tests
    - (*a*) 5 to 10 for each method.
4. Purpose of Test
    - (*a*) effect of interference—dissolved oxygen present.

Test No. 14:

1. Water Conditions
    - (*a*) city supply
    - (*b*) interference—28 ppm. sodium sulfite, and
    - (*c*) oxygen content—0.015 to 0.03 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
    - (*a*) preferred method.
3. Number of Tests
    - (*a*) 5 to 10 for each method.
4. Purpose of Test
    - (*a*) effect of interference—dissolved oxygen present.

Test No. 15:

1. Water Conditions
    - (*a*) city supply,
    - (*b*) interference—negligible, and
    - (*c*) oxygen content—0.0000 cu. cm. per liter.
2. Test Methods Employed—Starch Titration
    - (*a*) preferred method.
3. Number of Tests
    - (*a*) 20 to 40.
4. Purpose of Test
    - (*a*) establish precision of starch titration.

Test No. 16:

1. Water Conditions
    - (*a*) city supply,
    - (*b*) interference—negligible, and
    - (*c*) oxygen content—0.0000 cu. cm. per liter.
2. Test Methods Employed—Potentiometric Titration
    - (*a*) preferred method with 1 and 4 cu. cm. of each reagent.
3. Number of Tests
    - (*a*) 5 to 10 for each reagent quantity.
4. Purpose of Tests
    - (*a*) establish magnitude of reagent correction.

The personnel selected to conduct the tests included in the program were engineering graduates in training. The group was headed by a chemical engineer responsible for following the agenda as closely as practicable within the limitations of the apparatus. In addition, the supervisor was responsible for maintaining the techniques of the test procedure. The group consisted of three men headed by the supervisor, and were trained to use the apparatus prior to obtaining data for the evaluation. A fourth man was responsible for keeping the Oxygen Test Methods Committee informed as to the progress made. The members of the committee frequently visited the test site for the purpose of controlling the over-all program. Considerable effort was made to obtain a high degree of objectivity throughout the entire investigation.

TEST RESULTS

The averages of data for all tests are shown in Tables II, III and IV, and are analyzed for the purpose of comparison. Tables II and III show the

TABLE II.—AVERAGES OF DATA.

Test	Water	Test	Dissolved oxygen, cu. cm. per liter					Evaluation weight
			Estimated	Measured	Displacement	Measured 1 PE_m	Displacement plus 1 PE_m	
No. 1.....	City supply	Winkler	0.0000	−0.00090	−0.00090	±0.00068	−0.00158	3
		S.G. "A"	0.0000	−0.00090	−0.00090	±0.00069	−0.00159	2
		S.G. "B"	0.0000	−0.00142	−0.00142	±0.00139	−0.00281	1
		U. S. Navy Laboratory method	0.0000	−0.00006	−0.00006	±0.00066	−0.00072	4
No 3.....	City supply plus 2 ppm. ferric sulfate as iron	Winkler	0.0000	−0.00046	−0.00046	±0.00155	−0.00201	4
		S.G. "A"	0.0000	−0.00180	−0.00180	±0.00198	−0.00378	3
		S.G. "B"	0.0000	0.00274	0.00274	±0.00280	0.00554	2
		U. S. Navy Laboratory method	0.0000	0.00319	0.00319	±0.00244	0.00563	1
No. 4.....	City supply plus 1 ppm. ferrous sulfate as iron	Winkler	0.0000	−0.00013	−0.00013	±0.00195	−0.00208	2
		S.G. "A"	0.0000	−0.00002	−0.00002	±0.00135	−0.00137	4
		S.G. "B"	0.0000	−0.00372	−0.00372	±0.00136	−0.00518	1
		U. S. Navy Laboratory method	0.0000	−0.00011	−0.00011	±0.00141	−0.00152	3
No. 5.....	City supply plus 2 ppm. tannic acid as tannic acid	Winkler	0.0000	−0.00149	−0.00149	±0.00132	−0.00281	1
		S.G. "A"	0.0000	−0.00060	−0.00060	±0.00118	−0.00178	2
		S.G. "B"	0.0000	−0.00131	−0.00131	±0.00034	−0.00165	3
		U. S. Navy Laboratory method	0.0000	0.00040	0.00040	±0.00041	0.00081	4
No. 6.....	City supply plus 2 ppm. each ferric and ferrous sulfates as iron and 2 ppm. tannic acid as tannic acid	Winkler	0.0000	−0.00135	−0.00135	±0.00219	−0.00354	1
		S.G. "A"	0.0000	0.00026	0.00026	±0.00207	0.00233	2
		S.G. "B"	0.0000	−0.00038	−0.00038	±0.00193	−0.00231	3
		U. S. Navy Laboratory method	0.0000	0.00040	0.00040	±0.00073	0.00113	4

TABLE III.—AVERAGES OF DATA.

Test	Water	Test	Dissolved oxygen, cu. cm. per liter					Evaluation weight
			Measured	Estimated	Displacement	Measured 1 PE_m	Displacement plus 1 PE_m	
No. 2.....	City supply	Winkler	0.01656	0.01302	0.00354	±0.00125	0.00479	1
		S.G. "A"	0.01656	0.01835	−0.00179	±0.00216	−0.00395	2
		S.G. "B"	0.03810	0.03710	0.00100	±0.00114	0.00214	3
		U. S. Navy Laboratory method	0.01980	0.01915	0.00065	±0.00076	0.00141	4
No. 7.....	City supply plus 2 ppm. ferric sulfate as iron	Winkler	0.01460	0.01781	−0.00321	±0.00207	−0.00528	4
		S.G. "A"	0.01460	0.01975	−0.00515	±0.00153	−0.00668	3
		S.G. "B"	0.03110	0.04040	−0.00930	±0.00238	−0.01168	1
		U. S. Navy Laboratory method	0.02495	0.02001	0.00494	±0.00251	0.00745	2
No. 8.....	City supply plus 1 ppm. ferrous sulfate as iron	Winkler	0.01675	0.01560	0.00115	±0.00076	0.00191	4
		S.G. "A"	0.01675	0.01938	−0.00263	±0.00096	−0.00359	2
		S.G. "B"	0.03430	0.03140	0.00290	±0.00710	0.01000	1
		U. S. Navy Laboratory method	0.02050	0.01985	0.00065	±0.00185	0.00250	3
No. 9.....	City supply plus 2 ppm. tannic acid as tannic acid	Winkler	0.00936	0.00944	−0.00008	±0.00124	−0.00132	4
		S.G. "A"	0.00936	0.02004	−0.01068	±0.00139	−0.01207	2
		S.G. "B"	0.02780	0.04000	−0.01220	±0.00428	−0.01648	1
		U. S. Navy Laboratory method	0.01308	0.02000	−0.00692	±0.00084	−0.00776	3
No. 10....	City supply plus 2 ppm. each ferric and ferrous sulfates as iron and 2 ppm. tannic acid as tannic acid	Winkler	0.00600	0.01122	−0.00522	±0.00122	−0.00644	4
		S.G. "A"	0.00600	0.02000	−0.01400	±0.00121	−0.01521	2
		S.G. "B"	0.02440	0.04080	−0.01640	±0.00325	−0.01965	1
		U. S. Navy Laboratory method	0.00965	0.01990	−0.01025	±0.00134	−0.01159	3

results of the four test methods carried out under comparable conditions. Table IV shows the results obtained with the preferred method when tested in both the absence and presence of dissolved oxygen with sodium sulfite interference. It also includes results obtained with starch titration in the absence of oxygen and interfering compounds.

The condition of the water with respect to oxygen level, type of interference, and level of interference is indicated. The estimated oxygen level is based upon the assumption that the deaerator, because of its multistage design, was producing water of substantially zero oxygen content. The first stage of this deaerator consistently produced water deaerated to 0.002 cu. cm. per liter. The second stage of deaeration, which was added to the apparatus, reduced the oxygen level of the effluent to such a degree that it was not measurable by the test methods available.

The column headed "Estimated" is the assumed oxygen level of the water leaving the deaerator, and in the case of the high-level tests is the computed level based upon diluting the deaerated water with measured amounts of distilled water of known oxygen content.

TABLE IV.—RESULTS OBTAINED WITH PREFERRED METHOD.

Test	Water	Test	Dissolved oxygen, cu. cm. per liter				
			Estimated	Measured	Displacement	Measured 1 PE_m	Displacement plus 1 PE_m
No 11....	City supply plus 14.5 ppm. sodium sulfite as sulfite	U. S. Navy Laboratory method	0	0.00134	0.00134	±0.00082	0.00216
No. 12....	City supply plus 28 ppm. sodium sulfite as sulfite	U. S. Navy Laboratory method	0	0.00068	0.00068	±0.00172	0.00240
No. 13....	City supply plus 14.5 ppm. sodium sulfite as sulfite	U. S. Navy Laboratory method	0.02266	0.02035	−0.00231	±0.00071	0.00302
No. 14....	City supply plus 28 ppm. sodium sulfite as sulfite	U. S. Navy Laboratory method	0.02104	0.01880	−0.00224	±0.00181	−0.00405
No. 15....	City supply starch titration	U. S. Navy Laboratory method	0	0.00015	0.00015	±0.00153	0.00168

The column headed "Measured" represents the mean of the test results for the particular test method and test group.

The column headed "Displacement" is the difference between the mean of the estimated or computed dissolved oxygen in the samples and the mean of the test results for the same samples and is an indication of the accuracy of the test.

The column headed "1 PE_m" designates the first probable error about the mean of the test results included in the analysis.[4] Based upon statistical definition, 50 per cent of all readings will fall within the limits of the first probable error and 50 per cent will fall outside these limits. The first probable error is an indication of test precision.

The column headed "Displacement plus "1 PE_m" is the sum of the displacements and the first probable errors. The sign, plus or minus, shows the direction and magnitude of the expected maximum variation from the estimated or "true" oxygen level within the limits of the first probable error. The numerical value of the data in this column is an indication of the combined accuracy and precision of the test.

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[4] 1 PE_m = 0.6745 times standard deviation.

The column headed "Evaluation Weight" represents an arbitrary rank as to the value of the test for combined accuracy and precision. This was set up on a basis of assigning point values of 1, 2, 3, and 4 to the tests in each group, with 4 assigned to that test which shows the highest accuracy and precision, and 1 assigned to the lowest.

SIGNIFICANCE OF DATA

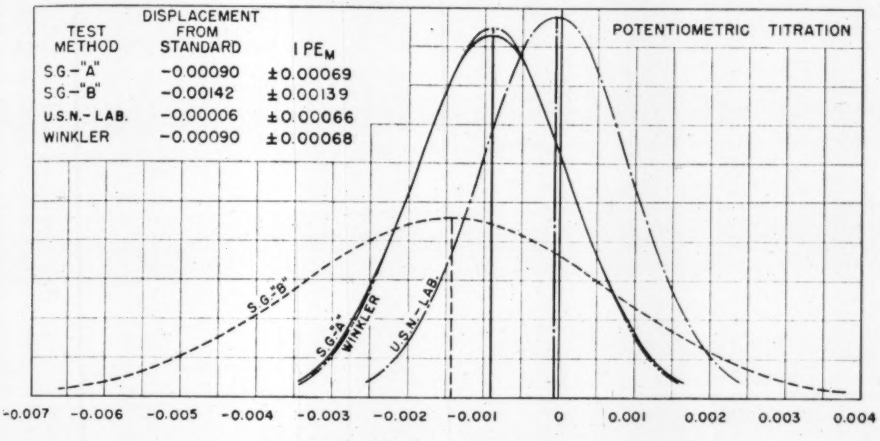
The tests conducted with city water with no interfering substances added and with a dissolved oxygen level approaching zero—Test No. 1, Table II—were for the purpose of establishing the basic accuracy and precision of the four methods under conditions closely paralleling that of a deaerator operating with boiler feedwater of reasonable purity. Many tests were conducted ranging from a minimum of 24 to a maximum of 56 for each method. It was found that when the data were plotted in the form of a histogram, the frequency distribution approached that of the theoretical normal curve. Bias was eliminated as much as possible in the selection of tests used in the analysis of setting up a criterion for the exclusion of data.

This criterion was based upon excluding only those tests which: (1) were known to have errors in the collection of the samples; (2) were known to have errors in the "fixing" of the samples; (3) were known to have errors in titration; (4) when the end point of titration occurred on the first 0.01-cu. cm. increment of thiosulfate addition; and (5) when the end point was indeterminate. Any test which could not be excluded on one of the above counts, regardless of its quantitative value, was included in the analysis.

In test No. 1 the Navy modification of the Schwartz-Gurney "A" test is in close agreement with the estimated level of oxygen and shows the remarkably low first probable error of ±0.00066 cu. cm. per liter. The Schwartz-Gurney "B" test shows the maximum displacement, reading low by 0.00142 cu. cm. per liter and shows a first probable error of ±0.00139 or about double that of the other tests included in the group. This difference in probable errors may be attributed to the fact that the effective sample size in the Schwartz-Gurney "B" test is one half of that for the other tests, thus a reduction in precision should be expected.

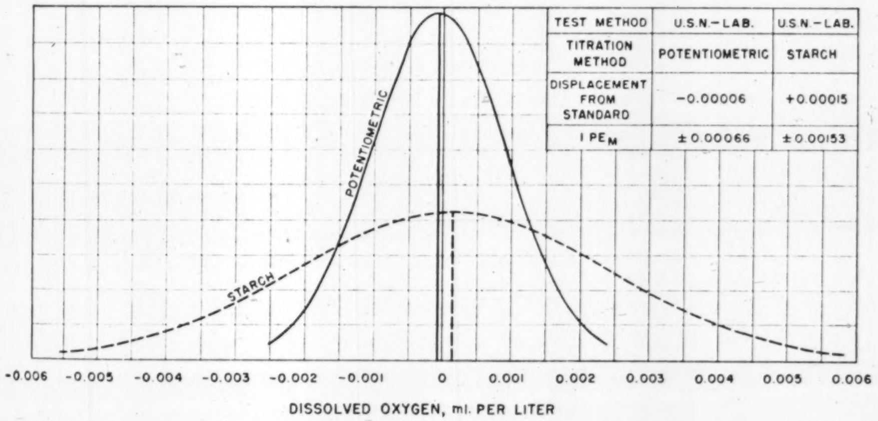
The normal distribution curves corrected to the same number of tests in each case (Fig. 5) show graphically the relative accuracies and precisions of the four methods of test under the conditions imposed in test No. 1. The acuity of the normal curves of the Winkler, Schwartz-Gurney "A," and Navy tests, as well as their relative displacement from the zero level, is significant of their accuracies and precisions as compared with the Schwartz-Gurney "B" method.

The significance of the first probable error in all other tests included in the first and second parts of the test program may be questionable, as the number of tests were too small (5 to 10) for unqualified statistical inference. The data for these tests are subject to bias in that if duplication of test results were obtained with a maximum variation of 0.003 cu. cm. per liter and all other requirements for inclusion were met, a minimum of five readings was considered sufficient for a true indication of accuracy. Some of these tests include only five test results, although it was necessary to double this number in many instances before a performance characteristic could be assumed as established. Therefore, the use of the probable error as indicative of the true precisions of these tests is subject to careful interpretation, and the assigning of a weighted value for evaluation purposes seemed to suggest itself. No attempt has been made at this writing to make such a modified evaluation, and the values of $1PE_m$ are used as recorded.



TEST METHOD	DISPLACEMENT FROM STANDARD	$1PE_M$
S.G.–"A"	−0.00090	±0.00069
S.G.–"B"	−0.00142	±0.00139
U.S.N.-LAB.	−0.00006	±0.00066
WINKLER	−0.00090	±0.00068

FIG. 5.—Relative Accuracies and Precisions of the Four Methods of Test in Test No. 1.



TEST METHOD	U.S.N.–LAB.	U.S.N.–LAB.
TITRATION METHOD	POTENTIOMETRIC	STARCH
DISPLACEMENT FROM STANDARD	−0.00006	+0.00015
$1PE_M$	±0.00066	±0.00153

FIG. 6.—Relationship Between Precisions and Accuracies of the Potentiometric *versus* the Starch Methods of Titration.

Reference is made to test No. 2, Table III, which is a comparison of the four methods under consideration with water containing an appreciable amount of dissolved oxygen. The Navy test again shows better agreement with the estimated dissolved oxygen level and a greater precision than do the other three test methods.

Tests Nos. 3, 4, 5, and 6 (Table II) show the comparative performance of the four test methods with various interfering compounds which were added to the water ahead of the deaerator. The estimated oxygen level in the samples approached zero. The displacement varies from a minimum of −0.00002 cu. cm. per liter for the Schwartz-Gurney "A" test with ferrous sulfate interference to −0.00372 cu. cm. per liter for the Schwartz-Gurney "B" test with ferrous sulfate interference.

It is significant that for all the test methods, conducted in the absence of dissolved oxygen both in the absence and presence of interference, a reasonable accuracy was maintained. The comparison of tests at high oxygen levels in the absence of interference (test No. 2 Table III) shows the Schwartz-Gurney "B" and the U. S. Navy Laboratory method as excellent from the point of view of accuracy, with the Winkler test and the Schwartz-Gurney "A" test somewhat poorer in performance.

Tests Nos. 7, 8, 9, and 10 (Table III) show the comparative performance of the four test methods with appreciable dissolved oxygen present in the sample and with interfering compounds, as noted, added to the inlet water supply ahead of the deaerator. The results indicate a serious reduction in accuracy, with the Winkler test showing the best over-all performance.

Reference is made to the variation in first probable errors of tests Nos. 3, 4, 5, and 6 (Table II) and tests Nos. 2, 7, 8, 9, and 10 shown in Table III. As previously stated, the number of individual tests conducted with each method in each test group was relatively few (5 to 10) as compared with the number of tests in test No. 1, Table II, which were deliberately expanded for the purpose of establishing precision. A comparison of the first probable errors for the nine short-run test groups shows that in five instances the Schwartz-Gurney "B" method had the greatest probable error, in three instances the Winkler method had the greatest probable error, in one instance the Navy Laboratory method had the greatest probable error, but in no case did the Schwartz-Gurney "A" method show the greatest variation in precision. This is in reasonable agreement with the precision rank of the Schwartz-Gurney "B" method when compared with the other tests as shown in test No. 1, Table II.

The Navy Laboratory method shows definite superiority in tests Nos. 1 and 2 where the condition of the water more nearly approaches that of normal boiler feedwaters. This same test when considered in its entirety shows a slight superiority over all others in tests Nos. 3, 4, 5, and 6. These latter test groups simulate a well deaerated but highly contaminated boiler feedwater requiring a most reliable test method for measuring dissolved oxygen content. They more nearly represent conditions to be met by a deaerator than the high-level interference tests. Therefore the Navy method was selected for further evaluation.

This evaluation was made with sodium sulfite interference at two concentrations both in the absence and in the presence of dissolved oxygen. As in all the other interference tests, the sodium sulfite was added to the water entering the deaerator. In addition, the accuracy and precision of the test with starch titration was determined in the absence of oxygen and of appreciable interference with a sufficient number of runs for comparison with test No. 1, Table II. The results are shown in Table IV.

The tests show a rather high order of correction for sulfite contamination which is of extreme importance in the testing of some condensates for dissolved oxygen. The agreement between accuracies of the Navy method conducted with potentiometric and starch titration is well within the limits of test precision. A comparison of test No. 15, Table IV, with the Navy test listed in test No. 1, Table II, shows a difference of 0.00021 cu. cm. per liter. A comparison of probable errors—±0.00066 with potentiometric titration and ±0.00153 with starch titration is indicative of the relative precisions of the two titration methods. Figure 6 shows graphically the relationship between the accuracies and precisions. The normal curves shown have been corrected to the same number of tests and the degree of acuity at the mean is significant of their relative precisions.

The tests conducted to determine the magnitude of the dissolved oxygen added with the reagents is in close agreement with the results reported by Adams, Barnett, and Keller (**4**). The value of 0.00728 cu. cm. per liter which they determined for 2 ml. of each reagent when added to a 500-ml. sample is very close to 0.00725 cu. cm. per liter as determined by the heat Exchange Institute tests. The strength of reagents used by the Heat Exchange Institute is the same as that specified in Method D 888–46 T[3] under "Referee Method." As stated in this method, this determination agrees very well with the work of White, Leland, and Button (**7**).

CONCLUSIONS

At the present writing, the Heat Exchange Institute Oxygen Test Methods Committee has made no recommendations relative to a preferred test method for endorsement by the Institute. Further analysis and correlation of the collected data will be required before the committee will be in a position to make a formal recommendation. Therefore, the conclusions which follow may not be in entire agreement with the final recommendations which will be submitted to the Institute for approval.

A classification of the relative importance of the areas of investigation may be made on the basis that most boiler feedwaters are reasonably pure. Therefore test validity is more important with the more commonly found purer waters than with the less commonly found highly contaminated boiler feedwaters. This classification is considered of primary importance in the evaluation.

Second in importance to test validity in pure waters is test validity at various oxygen levels. It seems reasonable to consider test accuracy and precision at extremely low levels of more importance than accuracy and precision at high dissolved oxygen levels.

The test methods investigated are listed as to their evaluated weight, as shown in Tables II and III. The

Test Method	Maximum Possible Evaluation Weight	Actual Evaluation Weight
CLASSIFICATION 1 Negligible Interference—Low Dissolved Oxygen Level		
U. S. Navy method	4	4
Winkler	4	3
Schwartz-Gurney "A"	4	2
Schwartz-Gurney "B"	4	1
CLASSIFICATION 2 Negligible Interference—High Dissolved Oxygen Level		
U. S. Navy method	4	4
Schwartz-Gurney "B"	4	3
Schwartz-Gurney "A"	4	2
Winkler	4	1
CLASSIFICATION 3 Abnormal Interference—Low Dissolved Oxygen Level		
U. S. Navy method	16	12
Schwartz-Gurney "A"	16	11
Schwartz-Gurney "B"	16	9
Winkler	16	8
CLASSIFICATION 4 Abnormal Interference—High Dissolved Oxygen Level		
Winkler	16	16
U. S. Navy method	16	11
Schwartz-Gurney "A"	16	9
Schwartz-Gurney "B"	16	4
CLASSIFICATION 5 Negligible and Abnormal Interference—Low and High Dissolved Oxygen Levels		
U. S. Navy method	40	31
Winkler	40	28
Schwartz-Gurney "A"	40	24
Schwartz-Gurney "B"	40	17

classifications are listed in the order of test-validity importance.

Upon inspection of the ratings of the various tests listed in the classifications, the Navy test shows superiority in all except classification 4. Its rank in classifications 1 and 2, the most significant area of investigation for application to normal boiler feedwaters as well as its slight superiority in classification 5, the over-all area under investigation, indicates that of the test methods compared the U. S. Navy modification of the Schwartz-Gurney "A" test is the logical selection for a single dissolved oxygen test method.

The effect of large quantities of interfering compounds upon the accuracy and precision of the test when used for oxygen determination in well-deaerated waters is not too serious in most instances, but for partially-deaerated waters, the imposing of limitations on the test seems desirable. In this latter instance, a rather complete water analysis may be required in order to interpret test results properly.

The use of a test requiring a reagent correction does not necessarily reduce accuracy. With the magnitude of the reagent correction so well established, there should be little doubt as to the rationality of its application. The degree of purity of reagent, as well as the technique employed in the addition of reagents to samples, may introduce errors. Such errors are normally within the control of the analyst.

With a test method employing an interference correcting sample, consistency in titration technique rather than titration method is the more important factor. The lack of precision of a starch titration may not necessarily reduce accuracy as compared to potentiometric methods if a sufficient number of tests are made to compensate for the wider variation in test results. Because of the inaccuracy of the starch end point, starch titration is not recommended for accurate determination of dissolved oxygen with test methods which do not employ an interference sample.

In order to obtain the accuracy and precision inherent in a test method, careful selection of glassware designed for ease in manipulation and for the elimination of error, as well as a carefully integrated and proven test and analytical procedure is of greatest importance. Precisions of approximately one part in one billion within the limitations of the first probable error, as shown in test No. 1, Table II, for three test methods cannot readily be duplicated with a poor selection of equipment and a questionable test procedure. Results of comparable consistency may be realized only by adhering to these requirements.

Experience gained during the investigation indicates that validity of sampling is to some degree a function of the provisions for sampling. Short, direct sampling lines with as few valves and fittings as necessary for safety and control are desirable. All joints must be absolutely tight, and the sample cooler adequate in size for cooling samples well below room temperature. An absolute minimum of rubber tubing should be used, and variation in the size of sampling line should be avoided.

The data presented represent a major portion of the work in the field of dissolved oxygen testing sponsored by the Heat Exchange Institute. The Oxygen Test Methods Committee found it necessary to limit the area of investigation to boiler feedwaters and to include only the more common interference. No attempt was made to develop any new test method or to evaluate the effect of interfering compounds on a quantitative basis. Rather, the intent was to determine the limitations of the test methods in general use. The immediate purpose of this investigation has, for the most part, been accomplished. However, much remains in the field of dissolved oxygen testing of boiler feedwaters for study and analysis.

Acknowledgments:

The author particularly desires to acknowledge gratitude to the Heat Exchange Institute which so generously sponsored this project, and to the Oxygen Test Committee which instigated the project and appointed the Oxygen Test Methods Committee to formulate the procedure and conduct the investigation.

The invaluable assistance of the members of the Oxygen Test Methods Committee—J. R. McDermet, Consulting Engineer; A. C. Dresher, Cochrane Corp.; F. Kemmer, Cochrane Corp.; J. L. Trout, Elliott Co.; and E. B. Kuhn, Elliott Co.—in establishing and supervising the project is appreciated.

The author also extends thanks to the Worthington Pump and Machinery Corp. for providing the test facilities and assigning personnel from its Research and Development Department to conduct the work. Much credit is due George Glover of Worthington for supervising and conducting the tests and to C. W. Hunter, also of Worthington, for assisting in the computations and analysis of data.

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DISCUSSION

MR. R. C. ADAMS[1] *(presented in written form)*.—The extensive work and the large amount of data reported in this paper deserve more than passing mention and quick compliments to the author. In compressing his report into the present limits some valuable information has been squeezed out and some of that given may have been distorted. I shall comment first on the methods used in obtaining the data and then on the treatment given the data itself.

The simple procedure for titration of iodine equivalent to the original dissolved oxygen, to which the name *Winkler* has been assigned, does not involve any correction for interference nor for dissolved oxygen in the reagents. That such a correction was employed in these tests is evidenced by the negative measured value from all "Winkler" tests on waters containing no dissolved oxygen, but the magnitude of the correction and the means by which it was determined are not stated. Similarly, the investigators made their determinations of dissolved oxygen in the fixing reagents at the end of their study, in test No. 16, presumably by a procedure similar to ours (**4**)[2]. What value for the reagent correction was employed before?

It seems appropriate at this time to urge the author and his sponsor, the Heat Exchange Institute, to abandon the awkward unit *cubic centimeters per liter* for dissolved oxygen and to adopt *parts per million* as has been done in the A.S.T.M. Methods D 596 – 46[3] and D 888 – 46 T[4]. It is incongruous to speak of a volume of oxygen gas (at standard temperature and pressure) dissolved in water of widely different temperature and pressure when the operator is concerned with the chemical equivalent of the mass of oxygen involved. The weight per weight unit, *parts per million*, is more convenient.

The paper should include, possibly as an appendix, an integrated and detailed description of the preferred procedure. Although this procedure was "in essential agreement with that described by the author in 1944," an analyst desiring to follow it must consult at least three sources of information: the author's paper mentioned (**6**), our 1943 paper (**4**), and the A.S.T.M. Method D 888. Such details, under the normal headings of *reagents*, *apparatus*, and *procedure*, would be very helpful to those subsequently seeking information. Incidently, they would advise, as the paper does not, how adjustment is made for the 5 to 10 per cent of the first one or two fixing-reagent additions which are trapped in the stopcock bore of the sampling flask and whether the end point of the electrometric titration is determined by locating the point of inflection in the thiosulfate *versus* millivolt curve or at the peak of the first differential of that curve.

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[1] Principal Chemical Engineer, Assistant Superintendent of Chemical Laboratory, U. S. Naval Engineering Experiment Station, Annapolis, Md.

[2] The boldface numbers in parentheses refer to the references appended to the Sebald paper, see p. 1137.

[3] Standard Method of Reporting Results of Analysis of Industrial Waters (D 594 – 46), 1946 Book of A.S.T.M. Standards, Part III-A, p. 480.

[4] Tentative Methods of Test for Dissolved Oxygen in Industrial Waters (D 888 – 46 T), 1946 Book of A.S.T.M. Standards, Part III-A, p. 1020.

The statistical treatment applied to the data suggests the undesirability of an engineer serving as his own statistician. While an awareness of statistical significance and a basic comprehension of statistical methods are helpful to many engineers, including myself, it will be found economical in data collected and more efficient in interpretation to consult a professional statistician in both planning an investigation and in analysis of the resulting data. I have been professionally advised that the data from the Heat Exchange Institute investigation, if they had originally been fitted into a "factorial design" could have been subjected to an analysis of variance with the resulting measurement of variations in results due to each method, each test, their interaction and the experimental error. The arbitrary ranking of the several methods would then have been unnecessary. Much of this information could still be salvaged by a competent statistician given access to the complete data from the investigation.

The use of *probable error* as a measure of variation is virtually obsolete; modern practitioners consider *standard deviation* to be more efficient. The symbol for probable error employed by the author is confusing. The subscript *m* usually designates the mean or arithmetic average but his explanation of the term indicates that it refers to the probable error of the individual results.

The author's discussion of the criteria used for exclusion of data raises some question as to the validity of the method used. The criteria listed are sound, with the possible exception of the fourth one. This one would apply only if the addition of a fixed amount of standard biniodate, or similar, solution were a regular part of the procedure. These criteria should be applied, however, for discarding a sample before calculation of a result. Any implication of selecting from results once calculated should be rigorously avoided.

In his final appraisal of the relative merits of the four methods, the author seems to have weighted the classification factors employed in his arbitrary rankings counter to their previously announced importance. His judgment of the comparative importance of the four factors, with which I agree, is that classifications 1 to 4 have that order of importance. However, in summing the weights from classifications 1 to 4 to obtain classification 5, classifications 3 and 4 have been given four times the weight of either classifications 1 or 2. If the four classifications are given equal weight, the best method appears to be at least $\frac{1}{3}$ better than the second; but if classifications 1 and 2 are given twice the weight of classifications 3 and 4, as seems reasonable from the author's discussion of the relative importance of these factors, the difference is even greater. Such a substitution for the author's classification 5, with "actual evaluation weight" converted to percentage of maximum obtainable score, yields the following comparison:

Method	Percentage Score
Navy (A.S.T.M. Referee)	91
Winkler	58
Schwartz-Gurney "A"	54
Schwartz-Gurney "B"	47

The paper reports the results of a careful and objective study by competent personnel. Much more information can be obtained from these results than has been made available. I congratulate the Heat Exchange Institute on their choice of the man who directed the study and compliment Mr. Sebald on his performance of the task assigned.

A. A. Berk[5].—The data presented would probably be of more use in the future if it were possible for the author to include with his findings the amount of ferrous iron and copper ordinarily present in the water from his deaerator. It might be of definite importance later when these results are compared to others.

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[5] U. S. Bureau of Mines, Eastern Experiment Station, College Park, Md.

MR. T. H. DAUGHERTY[6].—Now that a number of methods and techniques have been developed and employed in practice for the accurate determination of small amounts of dissolved oxygen, it is timely for a comparative study of them to be made. The author is to be commended for having made such a careful study. The ultimate aim of such work is the selection of the best method and the standardization of the technique for its operation so that the data obtained by any qualified investigator will be characteristic of the samples tested rather than of the method and the technique employed. Certainly it is true that individual variations in the technique for a given method have been more the rule than the exception.

All of the four methods compared are modifications of the original Winkler principle and three of them are based upon the Schwartz and Gurney principle of double titration to cancel out errors. Three of the methods require the subtraction of a pre-determined correction factor for dissolved oxygen and contamination in reagents and the fourth, the Schwartz-Gurney "B" method, internally cancels out these errors.

In general, the subtraction of a constant correction factor in a high precision method is objectionable, particularly where the correction factor is large in comparison to the determined value as occurs in the data reported here. However, as the author points out, three laboratories have now independently determined this correction factor and agree very closely. Under these circumstances, the use of such a factor does not necessarily reduce accuracy.

The Schwartz-Gurney "B" method, which internally compensates for dissolved oxygen and contamination and which does not require the subtraction of a correction factor, was effectively hindered in this work on the basis of sample size. It requires double the size of sample employed in the other methods in order to establish the same degree of precision. The use of double-size sample-collection flasks was deemed awkward and was avoided. With this decision, there appears to be but slight justification for including this method in the comparative study.

The variations among the results obtained with the four methods, which are chemically so closely related, in the presence of the several interfering substances added are puzzling. Undoubtedly further work will be necessary before these variations can be rationalized.

The comparative rating of the straight Winkler method, with apparently only the subtraction of the constant correction factor, is surprising in view of all previous experience, particularly in classification 1 (Negligible Interference—Low Dissolved Oxygen Level) and in classification 5 (Negligible and Abnormal Interference—Low and High Dissolved Oxygen Levels). If these results are substantiated in further work, the simplicity of collecting, fixing, and titrating only one sample will strongly recommend this method for many investigations where the greatest accuracy and precision are not required. The standardization of a simple and satisfactory control method is at least equal in importance to the standardization of a high precision referee method. With the possible exception of acceptance tests of equipment and of a very few specialized instances of corrosion at very low oxygen levels, it is far more important to know over a period of time with a minimum of effort and equipment whether or not the dissolved oxygen is consistently being reduced to uniformly low levels than it is to know at a given time whether the oxygen concentration is 0.003 ppm. or is 0.0003 ppm.

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[6] Research Chemist and Chemical Engineer, Hall Laboratories, Inc., Pittsburgh, Pa.

MR. R. C. ULMER[7] (*by letter*).—The Heat Exchange Institute is to be complimented for the very thorough manner in which it has undertaken the problem of evaluating methods for determining dissolved oxygen in water. The writer has given this problem considerable attention but was always stopped by the fact that zero oxygen water was not available. The writer is a little surprised at the author's reference to the city supply, that is tests Nos. 1 and 2, as having estimated zero oxygen or rather inferring that dissolved oxygen methods, such as the Winkler test, should give zero oxygen for such a water. This ordinarily would not be considered, owing to the presence of a rather large amount of impurities that might be expected to influence the results. Reference is made to the analyses of the water supply which show considerable organic ammonia, nitrite and nitrate present in the water. We wonder why consideration was not given to the use of a more pure water supply such for example as condensate, where these impurities would at least be at a minimum. In other words, it is a little difficult to interpret results, for example, upon the addition of nitrate when the water already contains a considerable amount of nitrate.

A survey of the data obtained indicates several very interesting facts, the foremost of which is the one that the preferred method came out ahead of the ordinary Winkler Method by only a slight if at all appreciable amount. In fact, as indicated by tests Nos. 7, 8, 9 and 10, the Winkler Method actually gave more accurate results than any of the other methods even though large amounts of contamination were present.

Owing to these apparent discrepancies it would appear wise to further consider the data. In this respect it often is a good idea to consider whether results are "reasonable."

Test No. 3.—The Winkler test actually shows less oxygen than the estimated value even though considerable ferric sulfate is present. The ferric sulfate of course would be expected to act the same as oxygen in which case the Winkler test should show results that are too high.

Test No. 6.—In some cases the method of deciding which test gives the best results is not always very obvious. For example, in the case of the Schwartz-Gurney "B" value for this test, the deviation was only 0.00038 cu. cm. per liter whereas the deviation for the U. S. Naval Laboratory Method was 0.00040, yet the evaluation weight is in favor of the U. S. Naval Laboratory method, which showed the greater deviation

Test No. 7.—Again the Winkler method shows a low value even though an oxidizing agent is present. The Winkler method was also the most accurate.

Test No. 8.—Here the Winkler method shows too high a value even though reducing agent or ferrous sulfate was present. Again the Winkler test was the most accurate.

Test No. 9.—Again the Winkler test was the most accurate even though a large amount of interference was present.

Test No. 10.—Again the Winkler test was the most accurate even though a large amount of interference was present.

Test No. 11.—The measured value for oxygen should be negative rather than positive if the sodium sulfite is causing interference.

Test No. 12.—The measured value for oxygen should be negative rather than positive if the sodium sulfite is causing interference.

Tests No. 13 and 14.—The rather large error indicates that the preferred method does not satisfactorily correct for sodium sulfite.

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[7] Technical Director. Industrial Dept., E. F. Drew and Co., Inc., New York, N. Y.

The apparent discrepancies of some of these results would lead one to believe that perhaps it really is not possible to evaluate a method for determining dissolved oxygen unless an oxygen-free water containing little if any contaminate is available. Certainly great progress has been made in setting up a test procedure and it is hoped that in the near future additional tests can be run possibly under the above referred to conditions.

MR. J. R. MCDERMET[8] (*by letter*).—This discussion relates generally to the criticisms which may be directed against the test procedures described in Mr. Sebald's paper. Some of them were anticipated by the sponsoring committee in advance of undertaking the work; others resulted from the investigation and as such might be called undesirable by-products.

All of the methods used represent some variation of the Winkler iodimetric method. Surveys of available methods made for this committee and also for a similar A.S.T.M. standardizing committee revealed that no other methods existed which were supported by any experience indicating any possibility of attaining comparable accuracy. Other methods were known which did not have the necessary accuracy. As of the present only two possible additional methods have been sugested. One of these, involving a colorimetric indicator, requires apparatus unwieldy to operate and transport and costly to acquire. The other is a titration method similar to the Winkler with a direct determination of manganese, and offers some promise of success if the thousands of dollars experimental expenses devoted to this paper were available to develop it. Reasonable prudence required limiting this testing program to the methods employed.

When a survey of existing literature on oxygen test methods is made, a very large amount of information turns up. Much of it is involved with technique highly individualistic with the experimenter, and detailed procedures are almost never carefully outlined. It is therefore almost a hopeless task to reconcile the data beyond using it to judge the general applicability to the proposed procedure. The author in his bibliography has given almost all the references to previous literature which were organized and conducted with sufficient care to be useful to others.

Criticism may also be directed against the selection of substances deliberately added to produce interference. In actual practice the cause of interference is obscure and involves such agents as the resins dissolved from wood tanks or paint films; decaying organic matter, etc. Analytical methods in water analysis are rather vague and result in the classification of many pollution agents by their symptoms in terms of some familiar chemical substance. The interferences added are typical of the substances, and the writer believes them to be an intelligent and useful selection although of necessity limited to a smaller number and type than is desirable.

There will be disappointment over the relatively poor showing of the Schwartz-Gurney "B" method. It has been widely used because of its ease of manipulation in spite of more awkward apparatus, and because it offered automatic compensation for the unavoidable addition of oxygen in the fixing reagents. Its failure to attain a more favorable showing is probably due to two reasons. One of them is mentioned by Mr. Sebald as more unfavorable sample size. The other—more probable in the opinion of the writer—is the different extent to which both interfering and fixing reactions proceed in different concentrations such as exist in sample and blank. Extreme dilution and instability of the interfering substances doubtlessly increase this limitation.

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[8] Consulting Engineer, Phila., Pa.

The U. S. Naval Laboratory method has one significant advantage. It provides a measured and adjusted iodine addition to react with interference in very acid, very alkaline, and substantially neutral solutions, and, in the case of low determined oxygen, of nearly identical concentration in both sample and blank. It has the disadvantage of requiring the subtraction of a correction factor for the dissolved oxygen in the fixing reagents. This correction has been evaluated by several investigators some of whom are cited by Mr. Sebald. The agreement between their values is excellent, and a higher accuracy and uniformity is afforded than will be required in test procedures. There is of course the possibility of one of the fixing reagents becoming contaminated in the course of use. This occasionally occurs, but fortunately it is impossible to have a sufficiently low contamination so that the test results do not show it in a glaring manner.

Mr. Sebald and his coworkers have made a definite improvement in the design of the McLean sampling flask. Their use of a micro pipette modeled after a hypodermic syringe will save a lot of sore fingers from manipulating a conventional pipette when a large number of determinations are to be made. It will simultaneously avoid the loss of many samples because of the more precise control possible.

The Heat Exchange Institute test code when it is later published will very likely be based upon a very explicit and very detailed method of procedure and an equally precise specification of apparatus and solutions required, all developed from usage many times repeated in this investigation. The accuracy attainable upon some types of samples may be more than that needed. The subsequent user will feel confident of the applicability of his own results. The necessities of abridged publication have handicapped Mr. Sebald in explaining how very well he has done his work; and unfortunately leave unanswered temporarily many questions pertinent to his reader who will wish to put the method to immediate use.

MR. J. F. SEBALD (*author's closure, by letter*).—Mr. A. A. Berke's comment is of considerable importance in judging the significance of the test data. Table I of the paper discloses the properties of the water used as the basis for all the tests and shows that the iron content varied from 0 to 0.07 ppm. Occasional checks of the water used at the test site were in substantial agreement with the water analysis given by the Passaic Valley Water Commission. No copper was reported by the Commission and further questioning of their chief chemist revealed that they have been unable to detect more than a trace of copper at any time.

Mr. Daugherty's considered discussion deserves more than passing comment and the Heat Exchange Institute is appreciative of its thoroughness.

Some explanation may be in order with respect to Mr. Daugherty's contention that the Schwartz-Gurney "B" method as described in the paper had such characteristics as to have but slight justification for inclusion in the comparative study. The Schwartz-Gurney "B" test is so devised that the effective sample size is approximately one-half of the volume of the largest sample. Sampling flasks over 500 ml. in volume are somewhat awkward to handle. As a result the volume customarily used in most oxygen test work is 500 ml. and was selected by the committee as the basis of volume for all tests under consideration.

Even though the effective sample size affects precision or the degree of duplication of results, it has little or no effect upon accuracy or the degree of agreement with the true value, provided that a sufficient number of test results are included in a given study. Since the evaluation was made on combined accuracy and precision there seems little reason to exclude the Schwartz-Gurney "B" test because of an expected handicap in precision. By following the same reasoning as propounded by Mr. Daugherty, the Winkler test might have been excluded because it does not include a procedure for compensating for interfering compounds in the water and therefore should perform poorly in the presence of interference. One of the objects of the study was to test those methods commonly in use, and the Schwartz-Gurney "B" test was considered by the committee to be in this category.

The variation in results of the high level oxygen interference tests, as questioned by Mr. Daugherty, may be partially explainable in that the chemical reactions in the presence of interference and other impurities are extremely complex. The electrometric end point of titration and the "dead stop" end point frequently become less definite with the highly contaminated waters and the precision of the tests in such cases will fall off. Since the project did not include a quantitative evaluation of interference, very heavy dosages of interfering compounds were used in an effort to expose the test limitations. Some inconsistencies in results as related to expected values have occurred. Undoubtedly a quantitative study would be helpful in rationalizing some of these results, but such a study was beyond the planned area of investigation. The work of White, Leland, and Button indicates that if a quantitative evaluation with interfering compounds were accomplished its interpretation would be extremely complex. For instance, in recognition of this fact they have recommended that a correction of 0.01 cu. cm. per liter be applied to oxygen values obtained when iron is present in quantities of from 0.7 to 5 ppm., primarily because there is no direct relation between accuracy of the test and the quantity of iron present in the water.

The results of the tests show that the Winkler test when properly conducted is a suitable instrument for the measurement of dissolved oxygen for the purpose of control at any of the levels investigated. It is not, however, suitable for the measurement of performance of deaerating equipment with an expected performance level of 0.005 cu. cm. per liter. In this, the author is in complete agreement with Mr. Daugherty, and although the purpose of the study was to determine a single most reliable method for referee purposes, the applicability of the Winkler test to the more general cases of dissolved oxygen measurement should not be overlooked.

The Heat Exchange Institute is grateful to Mr. Adams for the time and effort expended in his most thorough discussion.

The procedure for the Winkler test as used in this study involved a correction of 0.0072 cu. cm. per liter of dissolved oxygen added with reagents as well as potentiometric titration. While this procedure cannot be considered as standard with the test as originally devised by Winkler, present-day practice as generally applied to the basic Winkler test includes reagent correction as well as a correction for the starch end point based upon sensitivity at the temperature of titration. The use of potentiometric titration effectively overcomes the uncertainty of the starch end point. In the interest of accuracy and to permit a direct comparison of results, the committee thought it desirable to use the same equipment and procedures used in conducting the other tests.

The reagent correction which was questioned by Mr. Adams was determined by fixing a number of parallel samples of equal size with 4 ml. and 1 ml. of each of the reagents respectively and attributing the cause of the differences in results obtained to the effective difference in the amounts of reagents added.

The question of units for expressing dissolved oxygen was discussed with members of the Heat Exchange Institute because of their lack of agreement with units as parts per million as advocated by Mr. Adams. The unit of measurement which Mr. Adams refers to as an "awkward volumetric unit" is based upon solubility relationships as propounded by Charles and Dalton, and for the purpose of design or performance evaluation of any deaerating apparatus it is more significant and more readily usable than units of weight. Because engineers are generally familiar with the volumetric measurement of gases and because of the fundamental volumetric relationship of partial pressures to solubility, the Heat Exchange Institute considers it desirable to retain the volumetric unit of measure. For those who are more interested in the weight relationship, it is possible to convert the volumetric unit to the weight unit by multiplying by 1.4.

The intent of this paper was to disclose data which were obtained on test and not particularly to describe the detailed technique employed, although the presentation of the paper included a motion picture study illustrating the exact procedure used in conducting the tests with each step carefully explained. While it is agreed that the inclusion of the detailed procedure as an integral part of the paper would be helpful to an analyst interested in following the procedure used, the Heat Exchange Institute had planned to publish this information at a later date in much more complete form than could have been conveniently accomplished in the paper.

It is rather surprising that Mr. Adams considered the treatment of data as an attempt to make a complete statistical analysis, particularly in view of the fact that the paper clearly states that "further analysis and correlation of collected data will be required." The time available for the preparation of the paper, as well as the fact that the entire test program had not been completed at the time of writing, made it impossible as well as impractical to subject the data to such treatment. Competent advice from a member of the Statistical Department of a large eastern university contradicts the statements made by Mr. Adams relative to "factorial design" and complete analysis of variance. There is no question of "salvaging" the information as implied by Mr. Adams, and it seems probable that such a thought very likely originated from a misunderstanding of the test agenda and the tables disclosing averaged data.

It is agreed that the use of probable error as a measure of variance (not variation) is rapidly becoming in disfavor among statisticians, but it is still a valuable and useful measure of precision. Since the data were not analyzed by variance, which generally requires the use of standard deviation, probable error was used as the term more generally familiar to the non-statistician for the measurement of precision and because tables for this term and its significance are found in most engineering handbooks. Despite its unpopularity among statisticians, it is still used as a measure of precision in astronomy, geodesy, and artillary fire control. The symbol PE_m as used in the paper is carefully and accurately defined as the probable error about the mean. The test agenda shows a plurality of readings for each test group and it is evident that the probable error about the mean of each group of test results was used throughout the paper and not the probable error of the individual test results as suggested by Mr. Adams.

The fourth criterion for the exclusion of data, which Mr. Adams questioned, involves the addition of a measured amount of standard biniodate or similar iodine solution prior to titration. This procedure is necessary in order to perform the potentiometric titration with the tests employing interference samples at both high and low oxygen levels and with the Winkler tests at very low oxygen levels. It is sometimes necessary with the Winkler tests for higher oxygen levels, especially in the presence of large quantities of negative interference.

While the relative importance of the classifications of test application were listed and the premise upon which they were based defined, weighted values were not arbitrarily assigned because it was felt that for a qualitative evaluation a higher degree of objectivity would result by reporting the data as obtained. The assigning of weighted values will not change the order of placement of the tests in the classifications but will change their relative differences as evidenced by the revision of classification 5 as suggested by Mr. Adams. Such a procedure would be used in a quantitative evaluation but it is evident that it is likely to be dependent upon judgment for the assignment of arbitrary weights for the high and low oxygen level tests and involves as well an estimate of the significance frequency, type and level of interference expected to be found in the waters tested. Collection of data from the field and a review of existing available data to form a basis for such an evaluation is desirable. It would be interesting to know the premise upon which Mr. Adams based his selection of evaluation weights.

The Heat Exchange Institute is most appreciative of Mr. R. C. Ulmer's discussion. There are several points, however, which Mr. Ulmer has brought up which may be clarified by further discussion.

The low level dissolved oxygen tests were conducted with well-deaerated water approaching "zero" dissolved oxygen measured as oxygen. The author is somewhat surprised that Mr. Ulmer has inferred from the text of the paper that the Winkler test would be unaffected with respect to accuracy in the presence of possible interfering compounds in tests Nos. 1 and 2. It was recognized that any of the tests evaluated might be deficient under these and the other conditions listed—otherwise there would have been no need for the project.

Consideration was given to two additional water supplies other than the city supply actually used as described under "Selection of Test Site." The condensate available was highly contaminated with sodium sulfite, and the other available source, spray pond water, had a very high concentration of dissolved solids, including some sulfite. Had distilled water been used, involving considerable expenditure in additional equipment, adverse comment might have resulted on the basis that water of such purity would rarely be met in the field. The city supply was selected as the best medium available, being reasonably pure and yet sufficiently contaminated to bring into effect the reputed interference correcting qualities of the tests selected.

The fact that the Winkler test gave more accurate results in tests Nos. 7, 9, and 10 (the U. S. Navy Laboratory method gave more accurate results in test No. 8 but fell off in precision) can hardly be classified as an "apparent discrepancy." A review of the final report on "The Determination of Dissolved Oxygen in Boiler Feed Waters" submitted by Alfred H. White, Project No.

767, October 1937, for the Joint Research Committee on Boiler Feedwater Studies, shows that interfering compounds act irregularly and to a large degree unpredictably with the Winkler, Schwartz-Gurney "A" and Schwartz-Gurney "B" tests. On the basis of this report and the studies made by the Heat Exchange Institute it would not seem justifiable to classify lack of agreement with expected performance as an apparent discrepancy.

Mr. Ulmer has suggested that certain test results be inspected to consider whether they are "reasonable." These numbered tests commented upon by Mr. Ulmer are listed with the explanation of the results given for clarification.

Test No. 3.—Even though ferric sulfate interference was present, the level of interference as measured by interference samples under the same conditions was negative. The Winkler samples showed low results consistent with the direction of interference.

Test No. 6.—The evaluation weight assigned to the tests for selection on the basis of performance is explained in the paper as based on combined accuracy and precision and not on accuracy alone as Mr. Ulmer has assumed in his comment. Reference is made to test No. 6, Table III, which shows displacement plus PE_m to be -0.00231 for the Schwartz-Gurney "B" test and 0.00113 for the U. S. Navy Laboratory method with evaluation weights of 3 and 4, respectively.

Test No. 7.—Even though ferric sulfate interference was present, the interference level measured by interference samples under the same conditions showed the net interference effect as slightly negative. The Winkler samples showed low results consistent with the direction of interference.

Test No. 8.—Negative interference was present as measured by interference samples. While the Winkler samples showed high results inconsistent with the direction of interference, the magnitude of error from the estimated true dissolved oxygen level was 0.00115 cu. cm. per liter.

Tests Nos. 9 and 10.—The results of these tests were considered unreasonable by Mr. Ulmer because they showed less error with the Winkler tests than with the interference correcting tests. Complete objectivity would seem to preclude such a criterion in view of the data obtained and reported by White, Leland, and Button.

Tests Nos. 11 and 12.—The test method used in these cases was the U. S. Navy Laboratory method employing an interference sample. With the differential test employing an interference sample, the type of interference present whether positive or negative is no indication that test error should result in high or low readings. If the test is performed with full correction, the chances are equal that an individual result would be either high or low independent of the type of interference present. This is the basic principle incorporated in all three of the interference correcting test methods studied, and the fact that the particular test method employed in this case showed high results in the presence of a negatively interfering compound is no measure of inconsistency.

Tests Nos. 13 and 14.—Whether the preferred method yields a satisfactory correction for sodium sulfite interference is a matter of opinion. In the case of test No. 13, the interference measured as dissolved oxygen was -0.0417 cu. cm. per liter and the error was -0.00231 cu. cm. per liter, or 5.53 per cent of the interference level. In the case of test No. 14, the interference measured as dissolved oxygen was -0.1096 cu. cm. per liter and the error was -0.00224 cu. cm. per liter, or 2.04 per cent of the interference level. It would seem that errors of this magnitude while undesirable do not appreciably limit the application of this test method when used with sodium sulfite contaminated waters.

Of the ten tests cited by Mr. Ulmer as questionable, only one, test No. 8, shows an apparent discrepancy. However, the magnitude of this discrepancy approaches the accuracy limits of any of the test methods employed.

Mr. Ulmer questions the possibility of evaluating test methods unless oxygen-free water containing little or no contaminate is available but fails to mention his estimate of the permissible degree of contamination. Whether or not the conditions for testing agree with Mr. Ulmer's requirements for conducting an evaluation project of this type, the various tests considered showed sufficiently different performance under identical conditions to make the selection of a preferred method possible. Additional work of this general type is desirable, particularly with a view toward increasing the area of investigation.

The Heat Exchange Institute is indeed fortunate in having Mr. McDermet as a member of its Oxygen Test Methods Committee. His many years of experience with problems involving water characteristics, his familiarity with the principles of mechanical deaeration and their application to design and his comprehensive knowledge of dissolved oxygen measurement, all of which he gave generously in the interest of the committee's objective, were invaluable.

During the persuance of the program formulated by the Heat Exchange Institute, Mr. McDermet also served as chairman of a dissolved oxygen measurement standardizing committee for the A.S.T.M. To date there has been no interchange of data or private revealing of methods and procedures with the result that the two committees reached their conclusions entirely independently of one another. Mr. McDermet showed the highest degree of professionalism in having an intimate knowledge of the work of both committees and at the same time treating this information as confidential. The Heat Exchange Institute Oxygen Test Methods Committee is appreciative of having had the opportunity of working with Mr. McDermet as a helpful and contributing member.

The commentary prepared by Mr. McDermet is most welcome as it contributes background material and information which will be of considerable interest and importance to the reader. His discussion of the many problems considered in the formulation of the test program and his clear and concise description of the significant advantage of the U. S. Navy Laboratory method should foster confidence in the method for the potential user.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON INSULATING OIL

Committee D-9 on Electrical Insulating Materials is sponsoring a series of meetings through its Subcommittee IV on Liquid Insulation. The first of these meetings was held at Atlantic City, N. J., on October 16, 1946. The following papers were presented at this time:

Steam-Emulsion Number as an Index of Transformer Oil Serviceability—M. D. Baker
The Interfacial Tension Test and Its Significance in Appraising Performance of an Insulating Oil—G. W. Gerell
Application of the Interfacial Tension Test in Grading Transformers Relative to Serviceability—E. F. Walsh
Refresher on Statistical Analysis Applied to Two A.S.T.M. Oil Dielectric Strength Test Procedures—E. W. Greenfield

The first three of these papers were published in ASTM BULLETIN, No. 146, May, 1947, pages 90 to 100; the fourth, Mr. Greenfield's, paper is found in the ASTM BULLETIN, No. 149, December, 1947, p. 89.

At the Fiftieth Annual Meeting of the Society, held at Atlantic City, the second series of papers was presented June 17, 1947 as the Fourth Session of the meeting. Mr. L. B. Schofield, Commonwealth Edison, presided. This second Symposium was organized to bring together oil producers, apparatus manufacturers, utility engineers, and others to express their respective viewpoints on the two subjects selected for treatment at this session. This Symposium includes the following papers:

Advantages of an Inhibited Transformer Oil—T. E. Reamer and R. G. Larsen
Oxidation Inhibitors in Electrical Insulating Oils—Leo J. Berberich
Power Factor of Insulating Oils, Its Significance and Methods of Testing Stability—J. C. Balsbaugh
Serviceability Tests on Transformer Oil from the Viewpoint of the Maintenance Engineer—Frank J. Pohnan

These four papers are published in ASTM BULLETIN, No. 149, December, 1947, pages 58 to 89.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON SYNTHETIC LUBRICANTS

This Symposium on Synthetic Lubricants was presented at the Eighth Session of the 1947 Annual Meeting of the American Society for Testing Materials held in Atlantic City, N. J., June 18, 1947. The Symposium was sponsored by Section U-V on Instrument Oils, of Technical Committee B on Lubricating Oils, of A.S.T.M. Committee D-2 on Petroleum Products and Lubricants, through the efforts of E. H. Erck, Bendix Aviation Corp., Chairman of Section U-V, and J. C. Geniesse, Atlantic Refining Co.

The Symposium consists of the following papers:

"Ucon" Synthetic Lubricants and Hydraulic Fluids—J. M. Russ, Jr.
Synthetic Lubricants from Diesters—F. J. Glavis and H. R. Stringer
Synthetic Lubricants for Military Aircraft—C. C. Singleterry

Mr. Erck acted as Chairman for the session and Mr. Geniesse as the Secretary.

These papers together with the discussion have been issued by the A.S.T.M. as Special Technical Publication No. 77 entitled "Symposium on Synthetic Lubricants."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON RUBBER TESTING

The Symposium on Rubber Testing was held in Atlantic City, N. J., June 19, 1947 as the Twelfth and Thirteenth Sessions of the Fiftieth Annual Meeting. The Symposium was sponsored by Committee D-11 on Rubber and Rubber-Like Materials through a committee of the following men:

S. Collier, Johns-Manville Corp., Chairman;
H. E. Outcault, St. Joseph Lead Co., Vice-Chairman;
A. W. Carpenter, B. F. Goodrich Co.;
R. H. Taylor, National Bureau of Standards;
L. V. Cooper, Firestone Tire and Rubber Co.;
G. H. Swart, General Tire and Rubber Co.;
A. E. Juve, B. F. Goodrich Co.;
H. G. Bimmerman, E. I. du Pont de Nemours & Co., Inc.;
R. S. Havenhill, St. Joseph Lead Co.;
T. A. Werkenthin, Navy Department, Bureau of Ships;
W. P. Tyler, B. F. Goodrich Co.;
R. A. Schatzel, Rome Cable Corp.;
E G. Kimmich, Goodyear Tire and Rubber Co.;
A. R. Kemp, Bell Telephone Labs., Inc.;
R. D. Stiehler, National Bureau of Standards; and
J. J. Allen, Firestone Tire and Rubber Co.

The committee in charge of the Symposium arranged that it should cover the work that had been done with regard to the physical and chemical methods of test, both on synthetic and natural rubbers, making available to industry the accomplishments of Government and industry during the period of the war when secrecy had to be maintained.

The Symposium consists of the following papers:

Introduction—Simon Collier
The Significance of Voluntary Standards and Their Status in the Rubber Industry—Arthur W. Carpenter
Functions of Rubber Reserve, Past, Present, and Projected—W. R. Hucks
Development of Methods of Chemical Analysis of Synthetic Rubber—Willard P. Tyler
Developments and Improvements in Methods of Stress-Strain Testing—J. W. Schade
Development and Standardization of Tests for Evaluating Processibility—Rolla H. Taylor
Standardization of Testing and Inspection in Government Rubber Plants—Ludwig Meuser
Testing and Grading of Wild and Plantation Rubbers—Norman Bekkedahl
The Use of Statistical Methods in Rubber Evaluation—Marian M. Sandomire

Mr. Collier served as Chairman of the Twelfth Session and Mr. Outcault served as Chairman of the Thirteenth Session.

These papers together with discussion have been issued by the A.S.T.M. as Special Technical Publication No. 74 entitled, "Symposium on Rubber Testing."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON LOAD TESTS OF BEARING CAPACITY OF SOILS

At the opening session of the Fiftieth Annual Meeting of the American Society for Testing Materials held in Atlantic City, N. J. on June 16, 1947, the Symposium on Bearing Capacity of Soils was presented under the sponsorship of Committee D-18 on Soils for Engineering Purposes. The primary objective of this Symposium was to bring together in printed form all the factual data that could be accumulated on load test procedures. Organizations having sufficient information to warrant it were invited to prepare this information in the form of a paper to be presented at the Annual Meeting of the Society. Six papers were prepared and included in the Symposium:

Field Loading Tests for the Evaluation of the Wheel-Load Capacities of Airport Pavements—L. A. Palmer
Methods of Testing Soils for Runways and Foundations—Elwyn E. Seelye, W. D. Bailey, and S. D. Teetor
The Use of Load Tests in the Design of Flexible Pavements—W. H. Campen and J. R. Smith
Field Bearing Tests Applied to Pavement Design—Robert R. Philippe
A Cyclic Load-Test Procedure—Jean E. Hittle and W. H. Goetz
A Canadian Investigation of Load Testing Applied to Pavement Design—Norman W. McLeod

Mr. W. S. Housel, University of Michigan, presided as technical chairman at the session.

Another project of this Symposium Committee has been to compile a bibliography on load test procedures. The bibliography includes references previously assembled by Committee D-18 and is supplemented by the list of references supplied by the authors of the papers presented.

These papers together with the discussion and the extensive bibliography are issued as A.S.T.M. Special Technical Publication No. 79 entitled "Symposium on Load Tests of Bearing Capacity of Soils."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON EFFECT OF LOW TEMPERATURES ON MATERIALS

A Symposium on the Effects of Low Temperatures on the Properties of Materials was held in Philadelphia at the Franklin Institute on March 19, 1946. The Philadelphia District through a Program Committee headed by A. O. Schaefer, The Midvale Co., with G. E. Landt, Philadelphia Textile Finishers, Inc., and F. B. Foley, the Midvale Co. as Co-Chairmen, arranged to have papers presented by outstanding authorities covering plastics, rubber and rubber-like materials, and ferrous and non-ferrous materials, including weldments.

The Symposium consists of the following papers:

Low Temperature Behavior of Organic Plastics—G. E. Landt
Behavior of Elastomers at Low Temperatures—J. W. Liska
General Aspects of Effects of Subatmospheric Temperatures on the Properties of Metals —Maxwell Gensamer
Effects of Subatmospheric Temperatures on the Properties of Non-Ferrous Metals—Norman L. Mochel
Effects of Subatmospheric Temperatures on Weldments—Robert D. Stout
Low Temperature Behavior of Organic Plastics—H. K. Nason

These papers and their discussion will be published by the A.S.T.M. as Special Technical Publication No. 78 entitled "Symposium on Effect of Low Temperatures on Materials."

SUMMARY OF PROCEEDINGS OF THE PHILADELPHIA SPRING MEETING—SYMPOSIUM ON PAINT AND PAINT MATERIALS (1947)

The 1947 Spring Meeting of the Society was held in Philadelphia, Pa., at the Benjamin Franklin Hotel on Tuesday, February 25, 1947 in conjunction with A.S.T.M. Committee Week.

The meeting comprised a technical program entitled "Symposium on Paint and Paint Materials (1947)" supplementing an earlier Paint and Paint Materials Symposium held in Philadelphia in 1935 and another held on March 3, 1943, at the Buffalo Spring Meeting. The 1947 Symposium particularily emphasizes testing.

The present symposium was sponsored by Committee D-1 on Paint, Varnish, Lacquer, and Related Products together with the Philadelphia District Council. Mr. Carlton H. Rose, National Lead Co., was Chairman of the Symposium Committee. The Committee members were: P. O. Blackmore, Interchemical Corp.; C. C. Hipkins, Bell Telephone Labs., Inc.; L. A Melsheimer, United Color and Pigment Dept., Calco Chem. Div., Am. Cyanamid Co.; N. Van Loo, The Sherwin-Williams Co.; W. T. Pearce, Consultant on Organic Coatings (representing the Philadelphia District Council); and W. H. Gardner, Allied Chemical and Dye Corporation (representing the Administrative Committee on Papers and Publication).

The two sessions comprising the Symposium were held Tuesday morning and afternoon. Carlton H. Rose acted as over-all Chairman, with W. C. Hipkins, M. Van Loo, and L. A. Melsheimer serving as technical Chairmen.

The Symposium consists of the following papers:

Test Methods and the Paint Industry—John C. Moore
Methods of Evaluation of Industrial Finishes—R. A. Pringle and E. M. Yacko
Evaluation of Film Properties of Automotive and Appliance Finishes—Frederick G. Weed and Newell P. Beckwith
Methods of Evaluation of Metal Container Finishes—J. H. McKenzie
Methods of Evaluation of Marine Finishes—Allen L. Alexander
Particle Size by Gas Adsorption—Edmund N. Harvey, Jr.
Interchemical Inclined Tube Viscometer—Maynard R. Euverard
Stroboscopic Timer for Stormer Viscometer—E. P. Peterson and Joseph W. Prane
Introduction to Statistics—A. E. R. Westman
How Paint Technologists Can Use Statistical Methods—E. I. Stearns
Statistical Analysis of Test Data on Accelerated Weathering of Paints—Roy Hill, George S. Cook, and William E. Moyer

These papers together with discussion have been issued by the A.S.T.M. as Special Technical Publication No. 75 entitled "Symposium on Paint and Paint Materials."

Also in conjunction with Spring Committee Week the formal dedication of the new Society Headquarters at 1916 Race Street was held on Wednesday afternoon, February 26 at which time Past-President G. H. Clamer, on behalf of the members of A.S.T.M., presented the building to President

Arthur W. Carpenter. The formal dedication at the Society Headquarters was followed by a dedication dinner at the Benjamin Franklin Hotel sponsored by the Philadelphia District Council. The dinner featured greetings by the officials of the City of Philadelphia, University of Pennsylvania, Engineers' Club and The Franklin Institute. The principal address of the evening was given by Dr. E. U. Condon, Director of the National Bureau of Standards. Past-President T. G. Delbridge of the Atlantic Refining Co. acted as toastmaster.

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- Methods for the Determination of Soft Pieces in Aggregate—D. O. Woolf, 967. Discussion, 984.
- Report on Correlation of Laboratory and Service Abrasion Tests of Rubber Tire Treads—A. E. Juve, J. H. Fielding, and F. L. Graves. Published in ASTM BULLETIN, No. 146, May, 1947, p. 77.
- Testile Testing in Germany—H. F. Schiefer, L. Fourt, and R. T. Kropf. Published in ASTM BULLETIN, No. 144, January, 1947, p. 17.

Absorption Test.
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